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DOE/RL 88-27

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GROUT TREATMENT FACILITY DANGEROUS WASTE PERMIT APPLICATION



Reference herein to any trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof.

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Appendix	138	Grout	Treatment	Facility	Approval	of C	onstruction	າ
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1		ACRONYMS
2		
2 3 4 5 6 7 8 9 10 11 12 13	AASHTO ACI AEA AFFF ALARA ANSI API ARM ASME ASTM	American Association of State Highway and Transportation Officials American Concrete Institute Atomic Energy Act of 1954 aqueous film-forming foam as low as reasonably achievable American National Standards Institute American Petroleum Institute area radiation monitor American Society of Mechanical Engineers American Society for Testing and Materials
14 15	BED	building emergency director
16 17 18 19 20 21 22 23 24 25 26 27 28 29 31 32 33 33 33 33 33 33 33 33	CABF CAM CASS CC CERCLA CFR COE CRT DMF DMRHF DOE DOE-RL DOT DSSF DST DW EACT EC E/C ECC	Cochran's approximation to the Behrens-Fisher (t-test) continuous air monitor Computer Automated Surveillance System concentrated complexed Comprehensive Environmental Response, Compensation, and Liability Act of 1980 Code of Federal Regulations U.S. Army Corps of Engineers cathode ray tube Dry Materials Facility, formerly called DMRHF Dry Materials Receiving and Handling Facility U.S. Department of Energy U.S. Department of Energy U.S. Department of Transportation double-shell slurry feed double-shell tank dangerous waste Emergency Action Coordination Team (DOE-RL/EACT) equivalent concentration Engineer/Constructor Contractor Emergency Control Center
40 41 42 43 44 45 46 47 48 49 50	Ecology EDO EDTA EHW EMT EP EPA EP/APC EPDM FFTF FML	Washington State Department of Ecology emergency duty officer ethylenediaminetetraacetic acid extremely hazardous waste emergency medical technician extraction procedure U.S. Environmental Protection Agency emergency procedures and abnormal plant conditions ethylene-propylene diene monomer Fast Flux Test Facility flexible membrane liner

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1
         FTMS
                     federal test method standard
    2
         FY
                     fiscal year
    3
    4
         GR-CO
                     general radio-chemical operator
    5
         GTF
                     Grout Treatment Facility
    6
    7
         HDPE
                     high-density polyethylene
    8
                     Hanford Environmental Health Foundation
         HEHF
    9
         HELP
                     hydrologic evaluation of landfill performance (computer model)
  10
         HEPA
                     high-efficiency particulate air
  11
         HMRT
                     hazardous materials response team
  12
         HSWA
                     Hazardous and Solid Waste Amendments
  13
         HWVP
                     Hanford Waste Vitrification Plant
  14
         HVAC
                     heating, ventilating, and air conditioning
  15
  16
         IARC
                     International Agency for Research on Cancer
  17
         TC
                     ion chromatography
  18
         ICB0
                     International Conference of Building Officials
  19
         ICP
                     inductively coupled plasma
  20
C 21
         JRPT
                     junior radiation protection technologist
  22
C 23
         LA
                     analytical laboratory procedure
   24
         LCT
                     liquid-collection tank
15 25
         LDCRS
                     leachate detection/collection and removal system
         LR
                     laboratory reference (material specification procedure)
n _____
   27
  28
         MHSC
                     Medical and Health Services Contractor
   29
         MOU
                     Memorandum of Understanding
  30
         MSDS
                     material safety data sheet
  31
  32
         NA
                     not applicable
  33
         NCAW
                     neutralized current acid waste

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         NCRW
                     neutralized cladding removal waste
                     National Emission Standards for Hazardous Air Pollutants
  35
         NESHAP
  36
         NFPA
                     National Fire Protection Association
   37
         NO
                     nuclear operator
   38
         NPDFS
                     National Pollutant Discharge Elimination System
   39
         NPO
                     nuclear process operator
   40
         NRC
                     U.S. Nuclear Regulatory Commission
   41
                     nonradioactive compositionally representative
         NRCR
   42
                     National Sanitation Foundation
         NSF
  43
   44
         0EC
                     Operations and Engineering Contractor
  45
         OHP
                     Operational Health Physics
  46
         OJT
                     on-the-job training
  47
         ORM
                     other regulated material
  48
         OT
                     operator trainee
  49
  50
         PCA
                     Portland Cement Association
  51
         PFP
                     Plutonium Finishing Plant
  52
         На
                     negative logarithm of the hydrogen-ion concentration
```

```
1
      PIH
                  portable instrument house
2
      PISCES
                  Plant Instrumentation Surveillance Calibration Evaluation System
3
      PLC
                  programmable logic controller
4
      PSPL
                  Puget Sound Power and Light
5
      PSW
                  phosphate and sulfate waste
6
      PUREX
                  Plutonium/Uranium Extraction (Plant)
7
8
      0A
                  quality assurance
9
      OC.
                  quality control
10
11
      RAP
                  response action plan
12
      RCRA
                  Resource Conservation and Recovery Act of 1976
13
      RCW
                  Revised Code of Washington
14
      RDC
                  Research and Development Contractor
15
      RN
                  registered nurse
16
      ROD
                  record of decision
17
      RPT
                  radiation protection technologist
18
                  Superfund Amendments and Reauthorization Act of 1986
19
      SARA
20
      SCBA
                  self-contained breathing apparatus
                  senior radiation protection technologist
21
      SRPT
22
      SST
                  single-shell tank
23
      SWP
                  special work permit (clothing)
24
25
      TGE
                  Transportable Grout Equipment
      TGF
                  Transportable Grout Facility
26
27
      TOB
                  top of basalt
28
      TOC
                  total organic carbon
29
      TOX
                  total organic halogen
30
      TRU
                  transuranic (waste)
31
      TSD
                  treatment, storage, and/or disposal
32
33
      UHF
                  ultra-high frequency
34
35
      VHF
                  very-high frequency
36
37
      WAC
                  Washington Administrative Code
38
      WDOE
                  Washington Department of Ecology
39
      WESF
                  Waste Encapsulation and Storage Facility
                  water level
40
      WL
      WMA
41
                  Waste Management Area
                  Washington Nuclear Power (reactor name)
42
      WNP
                  Waste Receiving and Processing (Facility)
43
      WRAP
```

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```
ABBREVIATIONS
    2
3
4
5
6
          ac
                          alternating current
          Ci
                          curie
    7
8
          Ci/L
                          curies per liter
                          centimeter
          cm
    9
                          centimeters per second
          cm/s
          cm3
   10
                          cubic centimeters
   11
          °C
                          degrees centigrade
   12
   13
         d
                          day
   14
         dia
                         diameter
   15
   16
         e.g.
                          for example
   17
         et al.
                          and others
***· 18
         et seq.
                          and following
   19
  ~ 20
          °F
                         degrees Fahrenheit
C 21
         ft
                          foot
   22
                          feet per day
         ft/d
         ft<sup>3</sup>/s
cubic feet per second
   24
10 25
                         standard acceleration of free fall (gravity)
         g
   26
         gal
                         gallon
20 27
         gal/min
                         gallons per minute
   28
N 29
         h
                         hour
..... 30
         hp
                         horsepower
   31
 -- 32
         i.e.
                         that is
   33
         in.
                          inch
○34
   35
          km
                         kilometer
"" 36
         kVA
                         kilovoltampere
   37
         kW
                         kilowatt
   38
   39
                         liter
   40
         L/s
                         liters per second
   41
         1b
                         pound
   42
         1bf
                         pound force
         1bf/in<sup>2</sup>
   43
                         pound force per square inch
   44
         1b/h
                         pounds per hour
         1bm/ft<sup>3</sup>
   45
                         pound mass per cubic foot
   46
         1b/min
                         pounds per minute
   47
   48
                         meter
         m
   49
         mi
mi2
                         mile
   50
                         square mile
   51
         Mgal
                         million gallons
   52
         mil
                         mils
```

_		
1	min	minute
2	mL	milliliter
3	mo	month
4	Mrad	megarad
5	mR/h	milliroentgen per hour
Š	mrem/h	millirem per hour
7	•	
/	μ m	micrometer
8	m.s.l.	mean sea level
1 2 3 4 5 6 7 8 9		
10	p/b	parts per billion
11	P/ D	pares per bilition
	pCi/L	picocuries per liter
12		
13	r/min	revolutions per minute
14	.,	i di di di di di poi illittado
	_	
15	\$	second
16	stdft ³ /min	standard cubic feet per minute
17	·	•
18	V	volt
	٧	4010
19		
20	wk	week
21	wt%	weight percent
22		nergne percent
44		1
23	yd	yard
24	vr	vear

in

contract

1	
2	
3	
4	

APPENDIX 1

INTRODUCTION

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1 2		APPENDIX 1	
3			
4		INTRODUCTIO	N
5			
6			
/	1A	Notice-of-Deficiency Response Table	

APP

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GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> Ecology <u>Concurrence</u>

No. Comment/Response

142. Page 3C-1. The heat of hydration that will develop in the vault may raise the curing temperature above 90 degrees centigrade. These higher temperatures may have adverse effects on the solidification process. A discussion of how to mitigate this effect along with supporting justification must be provided before a permit can be issued. Response: The adiabatic calorimetry data discussed in the response to comment #29 will replace the short term transient thermal modeling of the grout vault to determine peak grout temperatures.

The adiabatic calorimetry data will result in conservative (high) values for the maximum grout temperature as it measures the grout temperature that would result if no heat was lost during the hydration process. Since some heat will be transferred out of the vault (conduction through walls, floor, and convection off surface), this will be a conservative (high) value for the maximum grout temperature. Text will remain unmodified.

- 143. Page 3C-2. GTF design and operations have changed significantly since this model was run. Therefore, the assumptions and parameters used should be reevaluated and the program rerun. Response: The assumptions used have been reviewed (except for those regarding heat of hydration) and were found to be conservative (resulting prediction of temperatures greater than expected). The heat of hydration portion of the modeling will be replaced by adiabatic calorimetry data. [p 3-25 through 3-28]
- 144. Page 4G-i. This information was not provided in April 1989. Please amend this date.

 Response: The design reports will be incorporated in the revised permit application and the referenced date will be deleted. [APP 4G]
- 145. Page 4H-3. Figure 4H-2 is missing. Please provide this figure.

 Response: Figure 4H-2 should not have been referenced. Reference to Figure 4H-2 will be deleted. [APP 4H, p 4H-3]
- 146. <u>Page 4I-i</u>. This information was not provided in April 1989. Please amend this date. Response: The design reports will be incorporated in the revised permit application and the referenced date will be deleted. [APP 4I]
- 147. <u>Page 4J-i</u>. This information was not provided in April 1989. Please amend this date. Response: The design reports will be incorporated in the revised permit application and the referenced date will be deleted. [APP 4J]

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GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> Ecology Concurrence

No.	 Comment/	<u>Response</u>

- 148 Page 5A2-4. How recent are these procedures? Is there a newer method available to analyze for nitrates other than the phenyldisulfonic method?

 Response: The procedures described in 5A2-4 were used in analyzing the sediments for the listed wells. The current method for analyzing nitrates in sampled sediments is by ion chromatography on soil extract. Text will remain unmodified.
- 149. Page 583-2. The water level in this well is averaged over 30 feet of screen. It is not satisfactory to compare these water levels to those of other wells with lesser screened intervals. Please address this issue. Response: The screen length was incorrectly listed as 30 feet and is only 20 feet in length. Other screens also are 20 feet. Text will be modified accordingly. [p 5-64, ln 14-52]
- 150. Page 5B3-9. The use of military time precludes the need for AM and PM designations. Please correct.

 Response: The am/pm designation will be deleted and Figures 5B-3.3 and 5B-3.5 will be modified. [p 5B3-10 and 5B3-14]
- 151. Page 583-9. What was the discharge rate after 400 minutes? Did this discharge rate change drastically? Response: The discharge rate varied during the test and affected the drawdown data. A plot of the variation in discharge and a brief discussion will be included in the text. [p 583-9, ln 4-18 and p 583-10 through 583-11]
- 152. <u>Page 5B3-14</u>. The date of pumping as listed in Figure 5B-3.4 should be from August 31 to September 1, 1987, and not 1978. Please correct. Response: Text will be modified. [p 5B3-8]
- 153. Page 5B3-14. It appears there is a possibility of delayed yield. A discussion of partial penetration effects should be included in the appendix text. Response: A discussion of these effects will be included in the text. [p 5B3-2 through 5B3-9]
- 154. Page 5C1-8. Typo. "Well 299-E25-32 is a single completion well." Should be "Well 299-E25-33 is a single completion well." Response: Text will be modified. [APP 5C, p 5C1-8]

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GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> Ecology Concurrence

No. Comment/Response

155. Page 5C1-13. The statement "The water is not turbid." is relative. What criteria is used to determine whether the water is turbid?

Response: The turbidity determination for this well at that time, as indicated in the geologic logs, was a qualitative evaluation. The current criteria for turbidity are ≤ 5 NTUs. To clarify the text, the sentence will be changed to read, "The water was visually determined to be non-turbid. Currently, wells are quantitatively considered to be non-turbid when they have been developed to ≤ NTUs." [APP 5C, p 5C1-14]

- 156. Page 5C1-14. Organic sampling will be conducted in the future. Therefore, wells must be constructed of materials agreeable to organic sampling.

 Response: Sentence will be modified and will state that the well construction material will be compatible with the sampled constituents. The current standard material used in well construction is stainless steel. [APP 5C, 5C1-14]
- 157. Page 5C2-2. The assumptions are not very realistic assuming a conservative approach. Are you trying to match conditions to the model, when the model should match the conditions? Response: The model will be rerun using a recharge rate to the vadose zone of 10 cm/yr as a more 'conservative' value. The results will be incorporated into the text. [APP 5C, p 5C2-5 through 5C2-20]
- 158. <u>Page 5C2-3</u>. There is a general breakdown in editing and checking the text in this section. The exponents are improperly written. Please correct.

 Response: Text will be modified accordingly. [APP 5C2 has been edited]
- 159. Page 5C2-17. Units for the "Waste Concentration" column must be provided. Response: Text will be modified accordingly. [APP 5C, p 5C2-13]
- 160. Page 5D1-1. If your sampling pumps are dedicated piston and submersible pumps, why do you use equipment for bladder pumps? Response: The bladder equipment was used as backup at one time. Since bladder pumps are no longer used, it will be deleted from the equipment list. [APP 5D, p 5D-1]
- 161. <u>Page 5D1-4</u>. Which wells have bladder pumps? Response: None. Text will remain unmodified.

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> Ecology Concurrence

- No. Comment/Response
- 162. Page 5D1-8. The accuracy should be listed as "+/- 0.01 ft" not just to "+ 0.01 ft". Response: The "-" sign will be added to the text. It should be noted that this number does not represent absolute accuracy, but the gradation to which the steel tape is read. [APP 5D, p 5D1-8]
- 163. Page 5D1-9. The first line repeats the last line of page 5D1-8. Please delete. Response: Text will be modified. [APP 5D, p 5D1-8]
- 164. Page 5D1-9. Steel tape method procedures should be repeated until two tape measurements agree within +/- 0.02 feet. In addition, the serial number or other identifying number of the measuring device should be recorded. Response: Text will be modified to read ± 0.02 feet, and will indicate that the measurement device identifying number should be recorded. [APP 5D, p 5D1-9]
- Page 5D1-12. The serial number or other identifying number of the conductivity meter should be recorded every time it is used.

 Response: Text will be modified to indicate that the conductivity meter identifying number should be recorded every time it is used. [APP 5D, p 5D1-11]
- 166. <u>Page 5D1-13</u>. Typo. "Jingle" should be "Single". "calibration" should be "calibrated". Response: Text will be modified. [APP 5D, p 5D1-13]
- 167. Page 5D1-14. Typo. "braking" should be "breaking". Response: Text will be modified. [APP 5D, p 5D1-14]
- 168. Page 5D1-17. Is U.S. Testing Co. the only laboratory planned to be used for analyzing these samples?

 Response: The U.S. Testing Co. is the only laboratory planned to be used for sample analysis, except for Tc-99. Pacific Northwest Laboratory will be used for analyzing Tc-99. Text will remain unmodified.
- 169. Page 5D1-22. Typo. "Tc04-" should be "Tc04-" and "HN03" should be "HN03". Response: Text will be modified. [APP 5D, p 5D1-21]
- 170. Page 5D2-5. The summation signs were left off of the equations. Please amend. Response: Text will be modified. [APP 5D, p 5D2-4]

9 0 1 1 7 8 5 9 0 1 7

GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> Ecology Concurrence

No. Comment/Response

- 171. Page 5D2-8. The first two lines of the page are repeats of the last two lines of the previous page. Please delete. Response: The repeated lines will be eliminated. [APP 5D, p 5D2-5]
- 172. Page 5D3-8. The conservative approach would be to control the false negatives rather than the false positives. It is more conservative to err on the side of the false positives. The statistical methods should be changed to accommodate this fact. Response: The overall false positive rate should be controlled on a facility-wide basis, rather than a well or parameter basis (McNichols and Davis 1988). One of the concerns associated with the use of CABF t-test method is that it does not adequately consider the number of comparisons that must be made (see Federal Register, Volume 53, No. 196, page 39720, October 11, 1988). The proposed CABF t-test procedure considers the number of comparisons that must be made [by replacing $(1 \alpha/2)$ by $(1 \alpha/2r)$ in a 'two-tailed' test and by replacing (1α) by $(1 \alpha/r)$ in a 'one-tailed' test where r = the total number of individual comparisons] in determining whether there is a statistically significant exceedance of background levels of specified chemical parameters and hazardous waste constituents.

It should be noted that for a given number of sample observations, Type I error (false positive) and Type II error (false negative) cannot be reduced at the same time.

To address the concern that the CABF t-test may result in 'false negatives', the following are implemented for the GTF.

- Currently two upgradient wells, 299-E25-25 and 299-E25-32, are in place. Another upgradient well, 299-E25-39, will be installed in 1990. These multiple upgradient wells will be used to estimate the spatial variability in the background levels.
- Proper analytical, quality control, and quality assurance procedures are established to reduce and control the measurement variability.
- Proper sampling equipment and techniques are used to control the errors due to sampling.
- The upgradient wells will be monitored for more than one year to establish background concentration levels which may need to be seasonally adjusted.

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> Ecology Concurrence

No.

Comment/Response

172. Reference (cont'd):

1) McNichols, R.J. and C.B. Davis, "Statistical Issues and Problems in Groundwater Detection Monitoring at Hazardous Waste Facilities," Fall 1988 Groundwater Monitoring Review, pages 135-150, 1988.

Text will remain unmodified.

- 173. <u>Page 8E-1</u>. Which of these courses, or which combination of courses, satisfies OSHA requirements requiring 40 hours of training for hazardous waste workers? (29 CFR 1910) Response: Tables will be modified to show OSHA requirements. [APP 8E]
- 174. Page 11A-i. This information was not provided in April 1989. Please amend this date. Response: The design reports will be incorporated in the revised permit application and the referenced date will be deleted. [APP 11A]
- 175. The QA/QC documentation will be required for all sampling and analysis activities. Please include a QA/QC plan.

 Response: The QA/QC plans covering all sampling and analytical work will be provided.

 [APP 3I and APP 5D4]

GROUT TREATMENT FACILITY

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> **EPA** Concurrence

NOD RESPONSE TABLE

No.

Comment/Response

EPA - Appendix 1, Section 4.4.4.2. The concrete composition for vault construction is not 1. specified.

EPA Recommendations: This section should specify Type II cement with tricalcium aluminate (C2Al) as indicated in Appendix 4E. This section should also specify concrete composition.

Air entrainment of 6 percent (more or less) should be considered in the concrete mix design to increase durability and moisture resistance. The proper amount should be verified through proper testing.

All aggregate used in the concrete should be alkali resistant. The following tests should be completed for aggregates to verify alkali resistance and chemical stability:

- *ASTM C 227 (mortar bar test)
- *ASTM C 289 (quick chemical test)
- *ASTM C 586 (rock cylinder test)

*ASTM C 150 - 84

Response: The concrete composition will be specified in the vault design report and construction specifications to be provided. [APP 41]

The composition does specify Type II cement, but does not specify tricalcium aluminate content. When tricalcium aluminate is not specified, typical Type II cement contains between 4 and 10 percent tricalcium aluminate. The only time it is necessary to specify tricalcium aluminate is if the sulfate concentration of either the makeup water or a solution that would normally come into contact with the concrete exceeds approximately 3,000 ppm. The typical waste to be processed at the GTF contains approximately 1-2 ppm sulfate. As a result, specification of sulfate-resistant Type II cement is not necessary.

The construction specification requires air entrainment of $5\% \pm 1\%$. The construction specification identifies American Concrete Institute (ACI) Standard 301 84-3 for all concrete construction activities. This standard specifies ASTM C 33 for identification and testing of aggregate materials which include the use of ASTM C 227, 289, and 586, as appropriate. Text will remain unmodified.

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No. _______ Comment/Response ______ Concurrence

2. EPA - Appendix 4E. The specification for concrete composition is incomplete.

<u>EPA Recommendation</u>: The concrete composition for vault construction should be specified completely as shown in Appendix 1, Section 4.4.4.2. Response: The concrete composition will be provided in the vault design report and construction specification. [APP 41]

3. <u>EPA - Appendix 4E</u>. The test report is not adequate. No basis is presented for using a simulated double-shell tank solution as a test solution rather than free liquid after grout reaction with actual waste material. 40 CFR 270.21(b)(1) and 264.301(a)(1)(i) require that liner-waste compatibility testing demonstrate that liner strength and performance are still adequate after exposure to waste leachates and to the waste.

<u>EPA Recommendations</u>: The concrete and reinforcing steel should be testing for compatibility with actual grouted waste and free liquid after the grout reacts with the mixed waste. After the grout reaction, free liquid will probably constitute the highest salt solution in contact with the concrete.

Compatibility tests should demonstrate that the concrete and reinforcing steel are not adversely affected by exposure to test samples under maximum design load and with maximum expected temperature, including heat generated by hydration of the grout matrix. Compatibility tests should include a margin of safety for the maximum expected temperature in case 90 °C is exceeded during hydration or afterward.

The impacts of surface drying and wetting of concrete and reinforcing steel should be evaluated.

The effects of the introduction of chemical impurities into the grout matrix from the addition of fly ash, blast furnace slag, or clays should be evaluated. These effects will be taken into account with test solutions consisting of free liquid after grout reaction.

Total organic carbon was not addressed in previous compatibility tests. The actual waste solution contains 3g/liter of total organic carbon and a number of inorganic constituents. Test solutions consisting of free liquid after grout reaction will take into account the effects of these constituents.

Response: A discussion of compatibility of the concrete and reinforcing steel will be included in the vault design report. [APP 4I]

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GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> EPA Concurrence

No. Comment/Response

3. Response (cont'd): The concrete must have at least short term compatibility with the tank waste, since the grout slurry properties are similar to the tank waste. The waste represents the worst case of chemical concentrations that could affect the concrete. The concrete should be compatible with the worst-case fluid that the vault might contain and in the case of process upsets, it is possible that waste might enter the vault.

The addition of grout formers to the waste buffers the pH from 12 to 14 down to 12 to 13. Therefore, the simulated tank waste is very representative of the grout slurry.

The grout formulation has been developed so there is no free liquid after several days of grout reaction, therefore, a representative free liquid is undefined.

If excess liquid is present during processing, it would be from water flushes of the process equipment and piping. It would be more diluted than the grout slurry and less aggressive to the concrete.

The disposal system is designed over the long-term to prevent percolating water from reaching the exterior of the vault or contacting the grouted waste. Therefore, the generation of waste leachates is unlikely, and such leachates would be less aggressive to the concrete than the simulated tank waste. If leachate were generated from the grout, it would likely be near equilibrium with calcium hydroxide in the grout and concrete at a pH of around 12 which would keep reinforced steel in the concrete passivated so it would not corrode. Text will remain unmodified.

4. <u>EPA - Appendix 1, Section 4.4.2.7</u>. The compatibility of grouted waste and free liquid after the grout reaction with the proposed asphalt liner have not been addressed. The Part B indicates that these tests are ongoing, and results of these tests will be presented in the revised Part B at a later date.

<u>EPA Recommendations</u>: Compatibility tests for the proposed asphalt liner should be completed in accordance with 40 CFR 270.21(b)(1) and 264.301(a)(1)(i).

The asphalt liner (at a specified thickness) on a concrete surface should be tested for compatibility with the grouted waste and free liquid after the grout reacts with the mixed waste. Any effects of total organic carbon and inorganic constituents should be addressed in the test results.

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> EPA Concurrence

No. Comment/Response

4. <u>EPA Recommendations (cont'd)</u>: Compatibility tests should demonstrate that the asphalt liner on concrete is not adversely affected by exposure to test samples under maximum and minimum hydraulic design conditions and with maximum expected temperature, including heat generated by hydration of the grout matrix. Compatibility tests should include a margin of safety for the maximum expected temperature in case 90 °C is exceeded during hydration or afterward.

Compatibility tests should demonstrate that the asphalt liner on concrete is not adversely affected by abrasion, which is expected to occur along the interior walls of the vault as the grout is flowing into and filling the vault. These tests should be conducted at the maximum expected temperature of the grouted waste, including some margin of safety greater than 90 °C.

Commercially available asphalt materials used for surface protection include at least two different products. Review of the properties of these two products indicates that both will soften and flow in the range of 85 to 120 °C and would not be suitable for use under a design condition of 90 to 100 °C. It may be possible that chemical additives can be added to the asphalt to prevent softening and flowing from occurring at maximum design temperatures.

Alternatives to the asphalt liner should be investigated. Alternate materials such as HDPE may be viable options for the interior of the disposal vault. Alternate lining systems will require careful consideration and pilot testing to overcome potential problems. One such potential problem is the high viscosity of the grout flow which could cause tearing of the liner system. Expansion and contraction of the liner material with a change in temperature is also a potential problem. In the case of HDPE which has a high coefficient of expansion, a change in temperature from 0 °C to 100 °C will expand the material 1 ft. in 100 ft. An liner or other synthetic liner will require an anchor system for support along the 34-ft. high vertical walls of the vault. In some cases, a batten anchor system can be used to anchor liner material to concrete. The batten anchor system consists of a series of stainless steel strips and bolts with neoprene washers. Compatibility testing of alternate liners with the grout-waste matrix and free liquid after grout reaction will be necessary. Pilot testing of the anchor system to a vertical concrete wall with grout flow at maximum design temperature should also be performed to guard against possible tearing of the liner material.

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O 1 1 7 3 5 5 0 2 GROUT TREATMENT FACILITY NOD RESPONSE TABLE

No. Comment/Response

EPA <u>Concurrence</u>

4. Response: Compatibility tests with the proposed asphalt-based liner have been conducted. The report will be included in the next permit application submittal. The tests were conducted with simulated waste. Simulated waste represents the most severe case for the liner, as free liquid and leachate would have a pH lower than the waste itself. See additional discussion in the response to comment 3. For data on leachate composition and results of EP toxicity tests refer to Serne (1989 - Leach and EP Toxicity Tests on Grouted Waste from Tank 106-AN).

If tests are conducted to determine the compatibility of the asphalt liner while attached to the concrete, the strength of the concrete would mask any property changes of the asphalt-based liner. Therefore, tests were conducted so that changes in the asphalt properties could be measured.

The simulated waste included organics in the compatibility testing that was performed. The total organic carbon was not monitored during the testing. Because the purpose of the liner is to reduce the possibility of drainage over several months before any excess liquid is removed from a vault, it was concluded that estimation of long-term impacts due to organics was not critical.

The compatibility tests on asphalt-based liners were to demonstrate that no severe degradation occurred over the 120 day duration of the test. They showed that significant changes do not occur with the selected material at up to 90 °C, which is greater than the liner should reach. (The maximum specification for the grout is 90 °C and if this temperature is reached, it would be at the center of the vault. The liner is expected to be several degrees lower than the peak grout temperature, so there is some margin of safety.) Separate engineering tests were conducted with the selected liner to demonstrate that the material did not flow at the proposed temperature and that it could span small cracks that might form in the concrete due to thermal stresses. These data are included in the engineering report. [APP 4K]

There is no credible mechanism for abrasion of the asphalt-based liner. There is a splash pad located where the grout slurry will hit the base of the vault. As the first grout enters the vault it will hit the splash pad and flow to the corners of the vault. The grout is very fluid (not like concrete), and at the low velocities it will not abrade the exposed liner on the floor. Further, because the grout gels rather rapidly, after approximately 30 minutes, the flow will occur on the grout surface instead of on the liner. There is no mechanism for shear at the walls.

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4. Response (cont'd): The asphalt-based liner that was selected does have chemical additives that prevent it from softening and flowing at the expected temperatures in the vault. Tests were conducted to confirm that there was not a flow problem.

Three types of asphalt liner were tested. In addition, alternative materials were tested for the catch basin liner. The grout is not 'high viscosity'; therefore, there is not a tearing problem due to the grout. Internal liners such as HDPE were considered, and were actually used in the vault that was used for unregulated waste. Due to construction difficulties, expansion/contraction problems and requirements to have the vault under slight vacuum, the internal plastic liner approach was abandoned. Secondly, from a failure standpoint, it is desirable to have different materials for primary and secondary containment. Text will remain unmodified.

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<u>EPA - Appendix 1, Section 4.4.3.1.2</u>. This section, which describes the leachate detection/ and collection and removal system does not clearly describe the HDPE and secondary liner system.

EPA Recommendation: The revised Part B application should provide greater detail regarding the lower liner system. The information available does not clearly describe how the HDPE liner will be protected from high point loading imposed by the gravel drainage media. A number of options should be considered to minimize point loading. For example, a layer of abraded rock smaller in size than the gravel drainage media could be placed on top of the HDPE liner to reduce point loading. A geotextile cushion fabric under the HDPE would also reduce point loading. All gravel materials used for the lower liner system must be sized to prevent plugging of the 4-in. perforated collection pipe. Response: A detailed description of the liner and the leachate detection/collection and

Test results showing the minimal impact caused by the point loading of the gravel drainage media on the HDPE will be provided. [APP 4H]

removal system will be provided in the vault design report.

6. <u>EPA - Appendix 4H</u>. The flexible membrane liner-waste compatibility test report is inadequate. No basis is presented for using a simulated double-shell tank solution as a test solution rather than free liquid after the grout reaction with the mixed waste material. 40 CFR 270.21(b)(1) and 264.301(a)(1)(i) require that liner-waste compatibility tests demonstrate that liner strength and performance are still adequate after exposure to waste leachates.

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90 5 **GROUT TREATMENT FACILITY**

NOD RESPONSE TABLE

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> **EPA** Concurrence

Comment/Response No.

EPA - Appendix 4H (cont'd): The test solutions used had a greater concentration of inorganic 6. salts than the actual double-shell tank solution. The test solutions also had no concentration of total organic carbon. However, the actual double-shell tank solution has 3g/liter of total organic carbon. Therefore, the data base is not adequate for evaluating the suitability of this liner material.

The effects of radiation exposure on the liner as reported is incomplete.

Test results of the effects of radiation exposure on the liner were reported only on the dimensional measurements.

EPA Recommendations: The 60-mil HDPE liner should be tested for compatibility with free liquid after grout reaction with actual mixed waste.

EPA Method 9090 compatibility test for wastes and membrane liners should be used in completing the tests. The test results also should address the effects of radiation pertaining to visual, tensile, and hardness aspects of the liner.

Compatibility tests should demonstrate that the 60-mil HDPE liner is not adversely affected by exposure to test samples under maximum design load and actual design conditions and with maximum expected temperature including heat generated by hydration of the grout matrix. Compatibility tests should include a margin of safety for the maximum expected temperature in case 90 °C is exceeded during hydration or afterward.

The effects of the introduction of chemical impurities into the grout matrix from the addition of fly ash, blast furnace slag, or clays should be evaluated. These effects will be taken into account with test solutions consisting of free liquid after grout reaction. Response: The report in the original permit application was not complete. A complete version will be part of the revised permit application. The basis for using the simulated waste is given in the report, and is described in the response to comment number 3. [APP 4H]

The test solution was the same as the reference composition used for developing the grout formulation.

Organic carbon was included in the test solution and was monitored at the end of each testing period. Total organic carbon in the test solution remained relatively constant.

No.

Comment/Response

6. Response (cont'd): Effects of radiation impacts on the tensile strength and hardness are reported and are included in the revised permit application along with visual observations. The tests showed that the small doses that the liner will receive on the exterior of the vault and in the catch basin will not affect its performance. In fact, the material should also be satisfactory inside the vault from a compatibility standpoint.

A summary of the results will be included in the revised permit application. [APP 4H]

The blast furnace slag, fly ash, and cement will lower the pH to the 12-13 range which is less aggressive to the HDPE. These components do not contain organics which may be detrimental to HDPE. Inorganics are not aggressive to HDPE, therefore, testing the less aggressive free liquid or leachate is not warranted.

7. <u>EPA - Appendix 1. Section 4.4.3.5</u>. This section on systems compatibility is not clear or complete concerning corrosion resistance of carbon steel components of the LDCRS system. Results of compatibility tests for carbon steel with this waste environment have not been provided.

<u>EPA Recommendations</u>: Carbon steel materials should be tested for compatibility with free liquid after grout reaction with actual mixed waste. The Chemical Engineering Handbook indicates that the usefulness of carbon steel in solutions containing NaOH, HaNo₃, or NaCl is limited due to expected corrosion rates.

With an NaOH solution greater than 50 percent, and with a temperature of 200 °F, the expected corrosion rate is greater than 0.05 in. per year. With an NaOH solution less than 50 percent and with a temperature of 200 °F, the expected corrosion rate is less than 0.02 in. per year. Proper test data should be provided to verify the stability of carbon steel in this environment.

Alternative materials to carbon steel should be considered for the leachate collection sump, pipe riser and connecting piping. Stainless steel and other materials should be considered and compatibility test data should be provided to verify its stability in this waste environment.

DOE/RL 88-27 Rev. 1, 01/17/90

APP A1-44

No.

ω· APP A1-45

Comment/Response

- 7. <u>EPA Appendix 1, Section 4.4.3.5 (cont'd)</u>: Corrosion protection for the LDCRS system should be verified. A cathodic protection system will require periodic maintenance that may be very difficult to perform and may not be adequate by itself for a long period of time. Protective coating materials should be considered. A section of the pipe riser above the high-liquid level of the sump also will be subject to a degree of both interior and exterior corrosion. Test data should be provided to verify the adequacy of all coating materials specified. Response: Substantial research and testing of the compatibility of double-shell tank waste solutions and carbon steel tank components has been performed at the Hanford Site. A report, "Prediction Equations for Corrosion Rates of A-537 and A-516 Steels in Double-Shell Slurry, Future PUREX, and Hanford Facilities Wastes" (PNL-5488), will be included in the revised permit application as an appendix. Further discussion is provided in the response to Ecology's comment 19.
 - <u>EPA General Comments</u> Regulations for landfills require that two or more liners and leachate collection systems be provided; one above the upper liner and one between such liners. If this double liner arrangement is not used then an alternate design must be employed that is at least as effective as the double liner arrangement. The liner system being designed for the grout waste disposal vaults includes an upper and lower liner but provides only one leachate collection system which is located between the liners. Should leachate leak through the vault walls or floor it will be contained and removed above the lower liner. However, the disposal system does not provide a backup leachate containment and collection system should the first one fail.

Using a buried concrete vault and catch basin as a disposal system for a grouted waste is a sound approach, and it is apparent that a substantial effort has gone into the conceptual design of the disposal system. At this time, however, the EPA has some concerns whether the current system meets the alternative design criteria stated in the regulations. Based on the information provided in the Part B permit application, an area of utmost concern and uncertainty is the asphalt liner on the inside surface of the vault. As pointed out in this report, a number of potential problems need to be addressed for any type of liner installed on the inside surface of the vault. The potential for free liquid inside the vault during the filling and curing periods is high. Also, filling of the vault could occur in stages due to disruption of grout mixing equipment, pumps, or piping. This could contribute to an increased amount of free liquid inside the vault. The behavior of a grouted waste can be complex and sometimes unpredictable for a waste mixture containing a substantial amount of organic constituents. This could also contribute to an increased amount of free liquid inside the vault.

GROUT TREATMENT FACILITY NOD RESPONSE TABLE

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> EPA <u>Concurrence</u>

No.

Comment/Response

- 8. <u>EPA General Comments (cont'd)</u>: Instead of trying to meet the alternative design criteria, another option would be to install another concrete catch basin and leachate collection system just below the catch basin presently being designed. This would fulfill the double liner requirement of the regulations.

 Response: Because the grouted waste is in liquid form when placed in the vault, it is constructed and operated as a surface impoundment, which requires two liners and one leachate collection system. Because of the unique nature of the waste, the contents of the surface impoundment solidify; therefore, the system is closed as a landfill. A detailed description of the liners and leachate detection/collection and removal system will be provided in the vault design report. [APP 41]
- 9. One specific area for which we could not find a reference in the Part B is how EPA's requirement for a "Response Action Plan" will be addressed. The Response Action Plan describes how the owner/operator will respond to leaks that reach the liner system's secondary leak detection system. The Federal Register, (May 29, 1987, vol. 52, no. 103, p. 20218) contains a proposed rule on this subject. The procedure in this proposed rule is being followed nationwide, until the final rule is issued. EPA Headquarters estimates that the final rule will not be finished for at least another year, and that it will not contain substantive changes from the proposed rule.

 Response: A 'Response Action Plan' will be provided in the revised permit application.

 [APP 7A]

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APPENDIX 2

FACILITY DESCRIPTION AND GENERAL PROVISIONS

2A Engineering Drawings 2B Hanford Site and Area Maps 2C Topographic Maps 2D Legal Description of the Grout Treatment Facility Property Boundary 2E Engineering ReportRoad Evaluation for Grout Treatment Facility		FACILITY DESCRIPTION
2B Hanford Site and Area Maps 2C Topographic Maps 2D Legal Description of the Grout Treatment Facility Property Boundary 2E Engineering ReportRoad Evaluation for Grout Treatment Facility		
2C Topographic Maps 2D Legal Description of the Grout Treatment Facility Property Boundary 2E Engineering ReportRoad Evaluation for Grout Treatment Facility	2A	Engineering Drawings
2D Legal Description of the Grout Treatment Facility Property Boundary 2E Engineering ReportRoad Evaluation for Grout Treatment Facility	2B	Hanford Site and Area Maps
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APPENDIX 2A

ENGINEERING DRAWINGS

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4		ENGINEERING DRAWINGS
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7	Appendix 2A	contains the following engineering drawings:
8		
9	H-2-76506	Instrumentation Engineering Flow Diagram
10	H-2-77596	Piping Plan
11	H-2-77635	Electrical Vault Plan 218-E-16-103
12	H-2-95889	Flow Diagram Transportable Grout Equipment Facility
13	H-2-95890	Flow Diagram Data Sheet Transportable Grout Equipment
14		Facility
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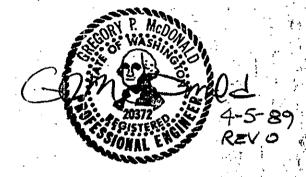
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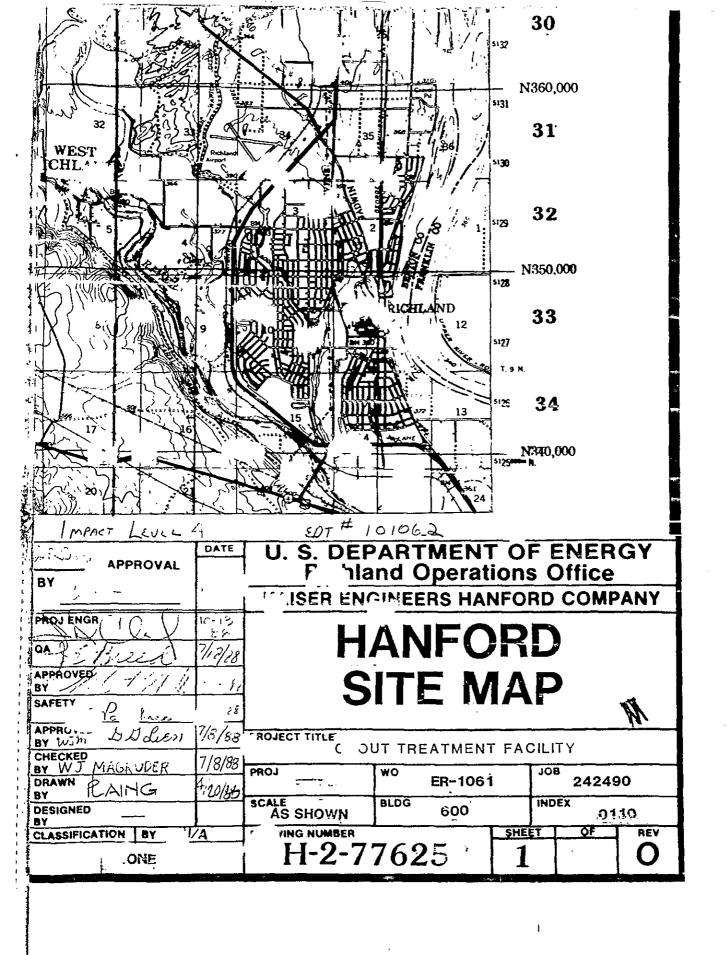


PLATE 2-1 TOPOGRAPHIC MAP

GROUT TREATMENT FACILITY BOUNDARIES

GROUT TREATMENT FACILITY BOUNDARY

241-AP-02D PUMP PIT



PLATE 2-2 TOPOGRAPHIC MAP

TRANSPORTABLE GROUT EQUIPMENT LAYOUT

LIQUID-COLLECTION TANK! MIXER MODULE

WATER SERVICE BUILDING TRUCK DISCHARGE HOOKUP

DRY-BLEND SYSTEM

ASPHALT

TRANSPORTABLE GROUT EQUIPMENT SITE

PLATE 2-3 TOPOGRAPHIC MAP

VAULT LAYOUT, FILLING AND CLOSURE SCHEDULE

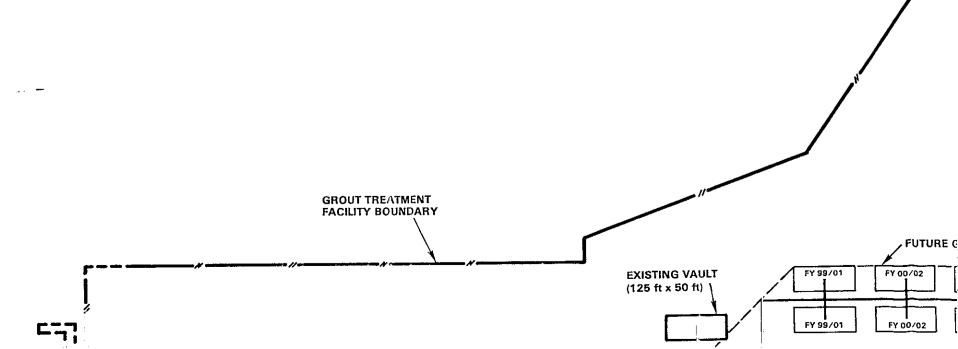
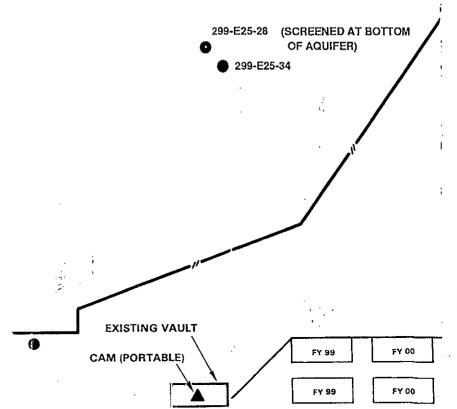


PLATE 2-4 TOPOGRAPHIC MAP

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PLATE 2-5 TOPOGRAPHIC MAP

TRAFFIC/SECURITY INFORMATION

TRANSPORTABLE GROUT EQUIPMENT SITE = SECTION COR (2" ALUMINUM PIPE / 3" CAP).

. = ALUMINUM ROD/3"CAP-3' OUTSIDE OF FENCE

O = NOT SET.

M = METERS

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		BY APPROVAL	DATE	U.S. DEPARTMENT OF ENERGY Richland Operations Office
	ļ	(1/1/clife	141182	KAISER ENGINEERS HANFORD COMPANY
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LEGAL DESCRIPTION OF THE GROUT TREATMENT FACILITY PROPERTY BOUNDARY

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APPENDIX 2D

LEGAL DESCRIPTION OF THE GROUT TREATMENT FACILITY PROPERTY BOUNDARY

Appendix 2D contains a certified legal description of the Grout Treatment Facility.

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APPENDIX 2E

ENGINEERING REPORT--

ROAD EVALUATION FOR THE GROUT TREATMENT FACILITY

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APP 2E-ii

ENGINEERING REPORT

Prepared for WESTINGHOUSE HANFORD COMPANY

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ROAD EVALUATION FOR GROUT TREATMENT FACILITY

October 1988

For the U.S. Department of Energy Contract DE-ACO6-87RL10900

Prepared by

KAISER ENGINEERS HANFORD COMPANY Richland, Washington

* KEH-88-30 ER9089

KAISER ENGINEERS HANFORD

KEH-88-30

ENGINEERING REPORT

FOR

ROAD EVALUATION FOR GROUT TREATMENT FACILITY



Westinghouse Hanford Company

Prepared by

KAISER ENGINEERS HANFORD COMPANY Richland, Washington

for

WESTINGHOUSE HANFORD COMPANY

W.J. Magnude Principal Lead Engineer	<u>9/29/88</u> Date	By McCleary Technical Documents	9/27/88 Øate
Safety	9-29-88 Date	Ain S. P. Ray Environmental	9/26/88 Date
Chief Design Engineer	<u>/0 -3-88</u> Date	Quality Assurance	10/3/88 Date
Project Manager	10-14-88 Date		
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ENGINEERING REPORT ROAD EVALUATION FOR GROUT TREATMENT FACILITY

I. INTRODUCTION

Major components of the Grout Treatment Facility (GTF) include the Transportable Grout Equipment (TGE) and waste management area. The waste management area includes underground vaults in which the grouted waste will be disposed of. A related facility, the Dry Materials Facility (DMF), is located approximately 1-1/4 miles to the west.

Construction, operation, maintenance, and monitoring for the 30-yr postclosure period requires vehicle access to the facility. This report evaluates the roads serving the GTF for both traffic volume and load carrying capability.

II. SUMMARY

The haul road route and disposal vaults access road examined in this report are adequate for the expected traffic. The haul road route handles the vehicle traffic for operation and maintenance of the Transportable Grout Equipment (TGE) and hauling of the dry materials. The disposal vaults access road is adequately sized to handle construction traffic required for maintenance and construction of the disposal vaults.

III. PURPOSE

Road access to the GTF must be maintained during its active life of 24 yr plus a 30-yr postclosure monitoring period. This report verifies that the roads are adequate to handle the volume and type of traffic expected.

IV. DESCRIPTION

Several State of Washington highways provide access to the Hanford Site from surrounding communities. These highways are designated as State Routes (SR).

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Numerous roads on the Hanford Site provide the necessary access for vehicles. Both the SR and roads on the Hanford Site are shown on figure 1.

Two separate routes are used to access the GTF and associated facilities. The first route examined in this report is within the confines of the 200-East Area and provides the required access to the DMF, TGE, and 241-AP Tank Farm. A second route located outside the 200-East boundary, but within the Hanford Site, provides access to the waste management area. These routes are designated as the haul road route (DMF to TGE) and disposal vaults access road.

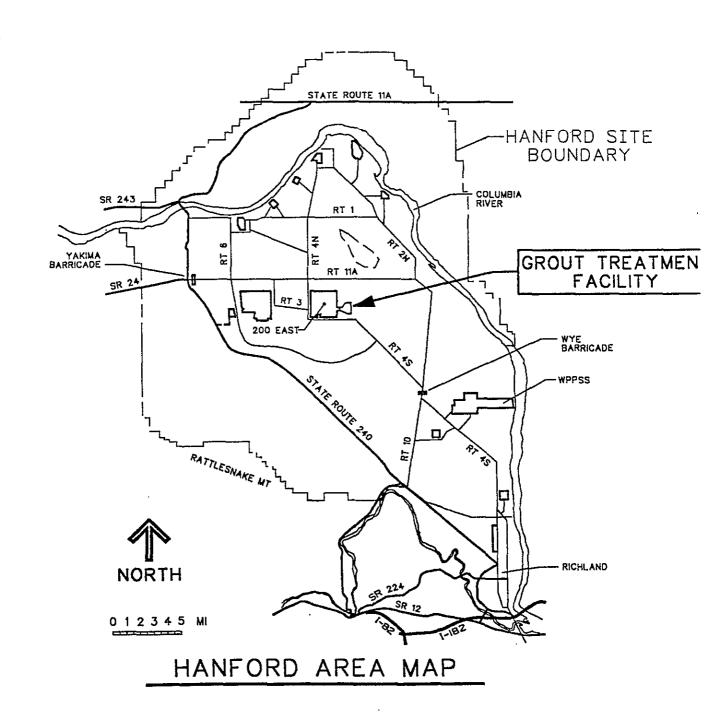
A. Haul Road Route (DMF to TGE)

The dry materials used in the grout mix are transported from the DMF to the TGE over the haul road route. The DMF, used for bulk storage of cement, flyash, and clay, is located near the center of the 200-East Area. Dry materials are mixed with the waste to form the grout at the TGE, which is located on the east boundary of the 200-East Area.

These dry materials are hauled by truck from the DMF to the TGE over existing roads. This haul route is from the DMF south to 4th Street, then east along 4th Street to Grout Drive located at the TGE as shown on engineering sketch ES-714-R2 (appendix A).

Access to the DMF from 4th Street was constructed in 1986. This road was designed to handle the expected car and truck traffic during operation of the GTF.

4th Street was built in the early 1940s during the initial construction of the Hanford project. This street has been maintained and is adequate for the haul road route.



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Figure 1.

- 3 -APP 2E-7 Grout Drive was also constructed during 1986 for the purpose of supplying access to the TGE. This road was designed to handle expected car and truck traffic during operation of the facility.

Other uses of the haul road route include vehicle access to the PUREX plant and to several tank farms located in the vicinity of the GTF. At present, this route has an approximate daily average traffic (ADT) count of 750 vehicles per day. A truck makes a round trip from the DMF to the TGE, hauling the dry materials every 2 hr during a grouting campaign. Two or three operators each shift and 20-30 maintenance and support vehicles require access to the TGE each day during grouting operations.

All haul road surfaces are paved with either an asphaltic concrete pavement or a bituminous surface treatment over an aggregate base. On roads where records are available, the pavement thickness is generally 0.20 ft thick over a 0.55 ft thick base.

Truck loading on the route is limited to HS 20-44 highway loadings as designated by the American Association of State Highway and Transportation Officials (AASHTO) (ref 1). This standard loading consists of a tractor truck with semitrailer. The entrance to the DMF and Grout Drive was designed to withstand this standard truck loading. Over the years, 4th Street has withstood truck and bus traffic without significant deterioration, and it is expected that it can withstand the truck loading during the hauling of dry materials.

Traffic control and signs on the haul road are in accordance with the Uniform Traffic Control Devices for Streets and Highways American National Standards Institute (ANSI) D6.1 (ref 2).

B. <u>Disposal Vaults Access Road</u>

Access to the disposal vaults will be from Route 4 South near the southeast corner of the 200-East Area. From the intersection of Route 4 South, this access road goes north approximately 2,000 ft on a 24-ft wide paved road. Next, the road goes east, then north into the GTF Boundary on a 24-ft wide gravel road. The final portion of this access route will lead down a ramp into the excavation required for vault construction.

Construction personnel and materials are primary users of this access route. Type and number of vehicles using this route will vary depending upon the amount of construction activity taking place.

Other vehicles using this route are security and maintenance personnel. Emergency access through a gate in the 200-East fence provides access to the TGE.

The paved road intersecting Route 4 South was paved during construction of 241-AP Tank Farm. It has experienced heavy construction traffic in the past and can be expected to withstand the traffic resulting from the GTF construction.

The gravel access road was part of the initial construction of Grout Facilities during 1987. With grading and other maintenance work, it is expected that this road will withstand the construction traffic.

Depending upon the amount of construction traffic, the ADT count may vary from 50 to 500 vehicles per day.

The final portion of this route, which provides a construction access ramp to the bottom of the excavation, is a 30-ft wide gravel roadway. Maximum grade will be 10%. As the excavation is enlarged for future vaults, this ramp will be relocated as required to provide construction access.

Truck loading on the access route is limited to HS 20-44 highway loading as designated by AASHTO. This standard consists of a tractor truck with semitrailer. The paved portion of this route

has withstood similar loading without significant deterioration. The gravel portion will be repaired if the road becomes rough and potholed.

Traffic control and signs on the access road are in accordance with the Uniform Traffic Control Devices for Streets and Highways (ANSI D6.1).

V. CONCLUSIONS

The haul road route and disposal vaults access road evaluated in this report are adequate for the projected traffic. Road maintenance will be required at regular intervals to provide access to the facility for a 24-yr active life and 30-yr post closure monitoring period.

VI. REFERENCES

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- Standard Specifications for Highway Bridges, 13th Edition, American Association of State Highway and Transportation Officials (AASHTO), 1983.
- 2. American National Standard Manual on Uniform Traffic Control Devices for Streets and Highways, ANSI D6.1-1978, w/Rev through Dec 1983.

APPENDIX A

Sketch

ES-714-R2

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APP 2E-12

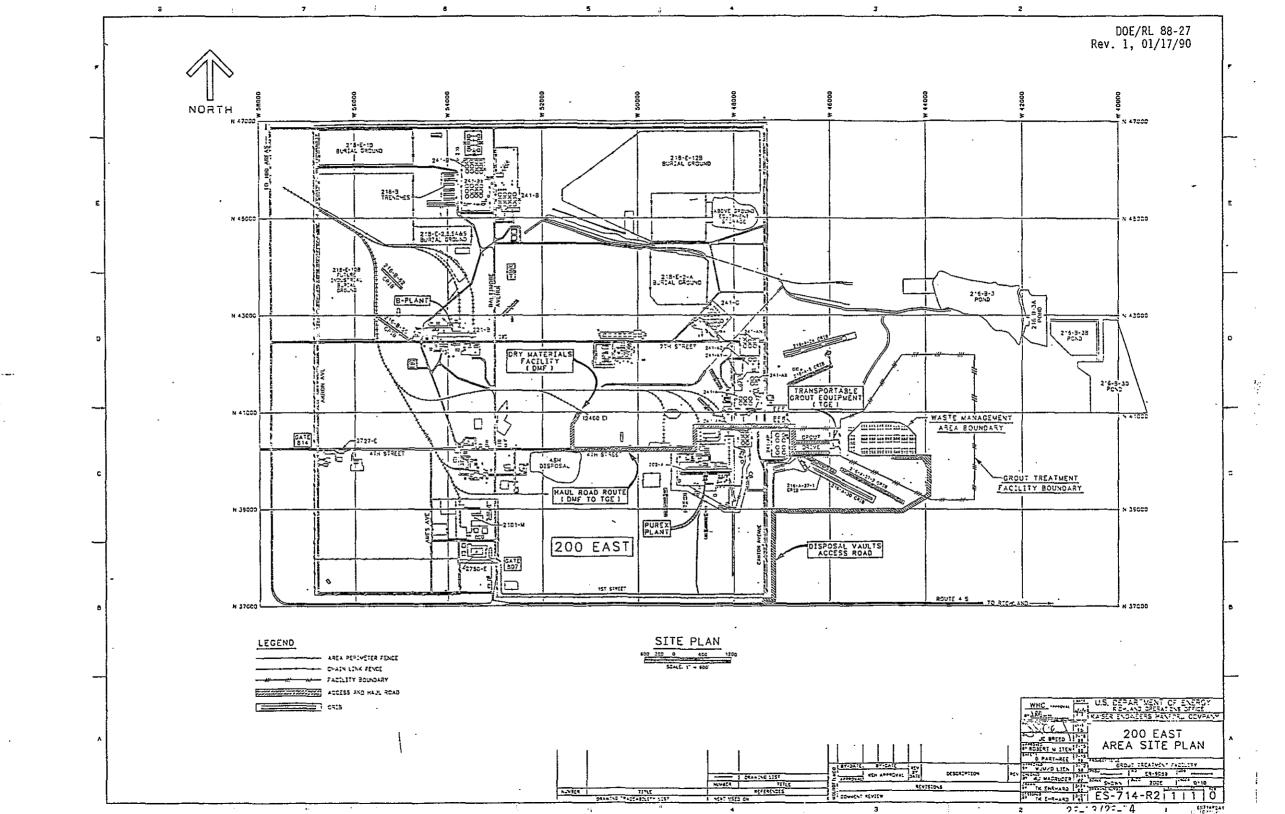
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APPENDIX 2F

DEPARTMENT OF ECOLOGY CERTIFICATE OF NON-DESIGNATION FOR CENTRALIA FLY ASH

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APP 2F-ii

DEPARTMENT OF ECOLOGY

CERTIFICATE OF NON-DESIGNATION

Certificate of Non-Designation Number:

84-3

Company Name and Address:

Pacific Power & Light Company and Others* Centralia Power Plant 913 Big Hanaford Plant Centralia, WA 98531

Telephone:

(206) 736-9901 -

Waste Status:

Undesignated:

Passes Criteria

Waste Description:

Process of Source of Waste:

Ply ash, bottom ash and slag waste, generated primarily from the combustion of sub-bituminous coal.

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Physical Nature:

Generation Rate:

Gray/Brown Solid

110,000 tons/month (maximum) 66,000 tons/month (average)

Type of Containers:

Mode of Transport:

Bulk

Truck to landfill to Widco Mine adjacent to facility.

Certificate Conditions - This Certificate of Non-Designation will be in force so long as the Centralia power plan burns coal from the Centralia mine on-site. Minor amounts of off-site coal will be permitted for blending purposes to achieve sulfur dioxide emission reduction. The on-site coal is classified sub-bituminous by ASTM D388, with average range of 7,600 to 8,200 BTU/pound.

Issued: June 19, 1984

Signature:

Minda L. Brothers Assistant Director

Office of Hazardous Substances and Air Quality Control

This Certificate of Designation is issued pursuant to WAC 173-303-075 and application for Certificate Number 84-3. The use of this Certificate to designate or not designate any waste other than that described in this Certificate and the applicant may be in violation of Chapter 173-303 WAC.

*See Attachment 1

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APP 2F-2

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APPENDIX 3

WASTE CHARACTERISTICS

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APPENDIX 3

WASTE CHARACTERISTICS			
3A	Double-Shell Tank Waste Compositional Modeling		
3B	Laboratory Analysis Reports for Double-Shell Tank Waste Stored in Tanks 241-AN-106, 241-AW-101, and 241-AN-103		
3C	Thermal Analysis in Support of Grout Temperature Limits and Heat-Loading Guidelines		
3D	Test Results for Extraction Procedure Toxicity Testing		
3E	Test Results for Toxicity Testing of Double-Shell Tank Grout		
3F	Pilot-Scale Test ReportEvaluation of Grout Processing Parameters		
3 G	Pilot-Scale Test ReportCorrelate Lab to Plant		
3H	Below-Liquid-Surface Supernatant Sampling Procedure for Underground Storage Tanks		
3 I	Laboratory Procedures and Grout 222-S/RCRA Laboratory Quality Assurance		

3J Grout Campaign Waste Composition Verification

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APPENDIX 3A

DOUBLE-SHELL TANK WASTE COMPOSITIONAL MODELING

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APP 3A-ii

APPENDIX 3A

DOUBLE-SHELL TANK WASTE COMPOSITIONAL MODELING

This appendix presents the basis for using the waste in tanks 241-AN-106, 241-AW-101, and 241-AN-103 to define a compositional range for double-shell tank (DST) waste. The utility of these wastes for defining a range is based on an analysis of waste origin data and projections that show a lack of any trend in DST waste sources that would add components not already found in inventory.

3A.1 WASTE ORIGIN

The available data on tanks 241-AN-106, 241-AW-101, and 241-AN-103 includes the history of waste in current inventory and laboratory analyses of samples taken from each of these tanks. Waste origin data correlates well with data from laboratory analyses.

3A.1.1 Basis for the Determination of Origin Data.

A history of DST waste in current inventory is acquired from computer files of tank farm transfer data. The transfer data are processed by a computer to follow waste entities throughout tank farms. The output from the computer program is a month-by-month listing of tank inventories in terms of volume and waste origins. Table 3A-1 presents an example of this output.

3A.1.2 Waste Origin Analysis.

An examination of waste origin data shows that the current DST waste in inventory is primarily older material dating before 1980. In fact, it appears that much of the chemical constituents in current inventory are from the salt well pumping program (residual liquid from retired single-shell tanks). Other wastes streams contributing to the inventory are either volumetrically small or dilute.

The waste in tank 241-AN-106 originates primarily from a phosphate waste stream from 100-N Area. The other waste in this tank comes from salt well liquid and minor amounts of dilute wastes from the facilities described in Chapter 2.0.

The waste in tank 241-AW-101 originates primarily from dilute wastes discharged from the Plutonium/Uranium Extraction (PUREX) Plant. The other waste in this tank comes from salt well liquid and minor amounts of dilute wastes from the facilities described in Chapter 2.0.

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Table 3A-1. Example Waste Origin Data.
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3
 4
             thous gal of DILUTE, COMPLEXED WASTE FROM B PLANT CESIUM PROCESSING thous gal of DILUTE, NON-COMPLEXED WASTE FROM B PLANT STRONTIUM PROCESSING
5
       10.9
 6
      358.6
             thous gal of DILUTE, COMPLEXED WASTE FROM B PLANT VESSEL CLEANOUT
7
       62.3
             thous gal of DILUTE, NON-COMPLEXED WASTE FROM B PLANT VESSEL CLEANOUT
      285.1
8
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM THE PFP (WITHOUT TRUEX)
9
       48.7
              thous gal of DILUTE, NON-COMPLEXED PUREX DECLADDING WASTE, THRU FY 86
    1,095.2
10
             thous gal of DILUTE, NON-COMPLEXED WASTE FROM PUREX MISC. STREAMS (NPR
    2,261.8
11
                                     FUEL),
12
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM S PLANT (222-S LABORATORY)
13
       57.0
             thous gal of DILUTE, NON-COMPLEXED WASTE FROM SINGLE-SHELL TANKS
      564.4
14
             thous gal of DILUTE, NON-COMPLEXED WASTE FROM T PLANT
15
      332.9
       80.4
             thous gal of DILUTE, PHOSPHATE WASTE FROM 100 N AREA
16
             thous gal of DILUTE, NON-COMPLEXED WASTE FROM 100 N AREA
      363.4
17
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM THE 300 & 400 AREAS
      164.3
18
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM UNC FUELS FABRICATION FACIL
19
       59.6
              thous gal of FLUSH WATER FROM MISCELLANEOUS SOURCES
20
    1,926.7
              thous gal of DILUTE, COMPLEXED WASTE FROM B PLANT CESIUM PROCESSING
21
       10.0
              thous gal of CONCENTRATED COMPLEX WASTE FROM EOFY 80 101AY INVENTORY
       47.7
22
              thous gal of CONCENTRATED COMPLEX WASTE FROM EOFY 82 102AZ INVENTORY
23
        6.4
             thous gal of CONCENTRATED PHOSPHATE WASTE IN EOFY 82 106AW INVENTORY
24
        7.4
              thous gal of DOUBLE-SHELL SLURRY FEED IN EOFY 82 101AW INVENTORY
25
      295.1
              thous gal of DILUTE DSSF FROM EOFY 82 102AW INVENTORY
26
        3.5
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM PRE-FY85 Z PLANT OPERATIONS
27
       65.8
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM FY82 100-N AREA WASTE
28
       47.1
29
                                     TRANSFER
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM B PLANT CESIUM PROCESSING
30
      140.0
              thous gal of DILUTE, PHOSPHATE WASTE FROM 231Z LABORATORIES
31
        1.5
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM PFP LABORATORIES
32
       29.4
              thous gal of DILUTE, NON-COMPLEXED WASTE FROM PRF PROCESSING
33
       88.4
      7.7 thous gal of DILUTE, NON-COMPLEXED WASTE FROM PFP RMC PROCESSING 170.1 thous gal of DILUTE, NON-COMPLEXED WASTE FROM EVAPORATOR PAD FLUSH
34
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```

The waste in tank 241-AN-103 originates primarily from salt well liquid. The other waste in this tank comes from other facilities described in Chapter 2.0.

3A.1.3 Laboratory Analyses of Double-Shell Tank Waste.

The laboratory analyses of samples taken from tanks 241-AN-106, 241-AW-101, and 241-AN-103 are given in Appendix 3B. A comparison of laboratory analyses with waste origin data (discussed previously) correlates well with the waste compositions given in process flowsheets.

The waste in tank 241-AN-106 has a much higher concentration of phosphates than the other two tanks. This is consistent with the fact that process flowsheets list the 100-N Area as the major source of phosphate discharged to the DSTs.

The waste in tank 241-AW-101 has a higher concentration of potassium than the other two tanks. This is consistent with process flowsheets that show PUREX as the major source of potassium discharged to the DSTs.

The waste in tank 241-AN-103 has a much higher concentration of aluminate than the other two tanks. The aluminate concentration identifies salt well liquids in the same way that potassium identifies PUREX waste and phosphate identifies 100-N waste. Process flowsheets show that most of the aluminate in DST waste comes from salt well liquids. The bounding case is near zero aluminate concentration when there is no salt well liquid in a tank.

3A.2 TRENDS IN FUTURE DOUBLE-SHELL TANK WASTE COMPONENTS

The determination of future DST waste character is based on compositions and volumes of the wastes as they are reported in process flowsheets. Table 3A-2 is a listing of waste streams (past, present, and future) contributing to future DST waste. A total of 14 major (by weight), soluble components are listed for these waste streams.

Using the reported volumes and compositions listed for DST waste projections, two sets of data were generated and graphed for the figures presented at the end of this appendix.

The upper graph on each figure represents the composition of one waste stream calculated at a reference salinity of 5M sodium. The sodium concentration is the reference component used for an approximate measure of overall salinity during the waste concentration process. It is also the predominant component in Hanford Site tank waste. The 5M sodium is chosen as a reference point for these graphs to emphasize that solubility issues have not been ignored. Operational experience indicates that the major components listed here precipitate significantly at concentrations above 5M sodium.

The data used to generate the lower graphic were calculated assuming that all of the waste streams generated within any one fiscal year are blended as a result of tank farm operations. The blending assumption represents DST-contributing waste streams mixed in a variety of combinations and volumes. The waste is concentrated to $5\underline{M}$ sodium and graphed. Since this is a material balance computation only, component concentrations are all proportional to the sodium concentration.

The data shown in the lower graphics of each figure are calculated endpoint concentrations for wastes that are received in that particular year (including 1986). In practice, the endpoint for the processing of these

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Table 3A-2. Waste Stream Numbering

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1: DILUTE, NON-COMPLEXED WASTE FROM B PLANT VESSEL CLEAN-OUT
2: DILUTE, NON-COMPLEXED WASTE FROM B PLANT CELL DRAINAGE
3: B PLANT AGING WASTE WUPERNATE FROM RETRIEVED AGING WASTE
4: DILUTE, NON-COMPLEXED WASTE FROM RETRIEVED COMPLEXED CONCENTRATE
5: DILUTE, NON-COMPLEXED WASTE FROM RETRIEVED PFP SOLIDS
6: DILUTE, NON-COMPLEXED WASTE FROM THE VITRIFICATION PLANT
7: DILUTE, NON-COMPLEXED WASTE FROM THE PFP (WITHOUT TRUEX)
8: DILUTE, NON-COMPLEXED WASTE FROM THE PFP (WITH TRUEX)
9: DILUTE, NON-COMPLEXED PUREX DECLADDING WASTE, THRU FY86
10: DILUTE, NON-COMPLEXED WASTE PUREX DECLADDING WASTE, FY 1987 ON
11: DILUTE, NON-COMPLEXED WASTE FROM PUREX MISC. STREAMS (NPR FUEL) FY86
12: DILUTE, NON-COMPLEXED WASTE FROM PUREX MISC. STREAMS (NPR FUEL), FY87 ON
13: DILUTE, NON-COMPLEXED WASTE FROM SHEAR/LEACH PROCESSING OF NPR FUEL
14: DILUTE, NON-COMPLEXED WASTE FROM PUREX MISC. STREAMS (FFTF)
15: DILUTE, NON-COMPLEXED WASTE FROM S PLANT (222-S LABORATORY)
16: DILUTE, NON-COMPLEXED WASTE FROM SINGLE-SHELL TANKS
17: DILUTE, NON-COMPLEXED WASTE FROM T PLANT
18: DILUTE, NON-COMPLEXED WASTE FROM 100 N AREA
19: DILUTE, NON-COMPLEXED WASTE FROM THE 300 AND 400 AREAS
20: DILUTE, NON-COMPLEXED WASTE FROM UNC FUELS FABRIFICATION FACILITY
```

Note: A more detailed explanation of these facilities is given in Chapter 2.0.

wastes is reached at later points in time than is shown on these graphs. The wastes received in 1986, for example, may not be completely evaporated until the end of 1988 or beyond.

3A.2.3 The Composition of Newly Generated Waste.

There are three major differences between the laboratory analyses presented in Appendix 3B and the typical future waste deduced from the lower graph on each figure. Two of these exceptions can be explained as the difference between wastes that have been generated in the past and newly generated waste.

The ratio of nitrate to nitrite in laboratory analyses of current DST waste inventory is approximately 1:1, while calculations show that the ratio will be much greater than that for typical newly generated waste. The difference in nitrate/nitrite ratios correlates readily with the analysis of inventory history. As waste ages, the radioalysis of nitrate will result in nitrite.

A second difference between laboratory analyses and calculated compositions is the aluminate concentration. The laboratory analyses all show aluminate concentrations in the 0.4 to 0.7 $\underline{\text{M}}$ range at 5 $\underline{\text{M}}$ sodium. The calculated average is less than 0.3 $\underline{\text{M}}$. The difference correlates readily

with the analysis of inventory history. Inventory history shows that the origin of DST waste is largely salt well liquids. Salt well liquids have high concentrations of aluminate from the dissolution of aluminum-clad fuel rods. The large-scale dissolution of aluminum cladding is no longer typical at the Hanford Site.

A third difference between laboratory analyses and calculated composition is the concentration of chloride. Calculations show a high chloride concentration of 0.02M. Sample data show chloride as high as 0.1M. Additional investigation is required to determine the reason for this discrepancy.

3A.2.4 Significant Components.

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The waste composition graphics show that most of the significant 17 components in tank farm wastes are not unique to any one waste stream. For example, tank farm specifications require the presence of hydroxide and nitrite in every waste stream with few exceptions. For these common components, the year-to-year variations have only a minor influence in the 21 determination of a concentration range.

For some components, there is a significant difference in concentration from year to year. These differences are attributed to the influence of one of a few of the significant waste streams.

28 3A.2.5 Significant Waste Streams.

A review of the data presented in Table 3A-2 and in each figure shows that three waste streams have a large influence on the chemical content of DST waste. Other waste streams are small, dilute, or essentially have no unique 33, components. The presence of large amounts of potassium and fluoride correlates with the generation of PUREX waste. An increase in nitrate concentrations correlates with projected complexant pretreatment operations. An increase in nitrite and aluminate concentrations correlates with an increase in salt-well pumping. The figures also show that high phosphate concentrations, prevalent in 241-AN-106 waste, are not expected in future DST waste.

3A.2.6 Synthetic Waste Used for Evaluations of Double-Shell Tank Grout.

Even though the blend of waste origins in the current inventory of DST waste is not representative of all future blend ratios, the concentration of components in the samples generally is consistent with calculations of future DST waste. The synthetic waste used for preliminary evaluations of the grout process is based on laboratory analysis and is a nearly perfect representation of the nominal, calculated DST waste composition, as shown on the graphics at the end of this appendix.

APP 3A-5

7.

3A.3 APPLICABILITY TO OTHER WASTE MANAGEMENT SCENARIOS

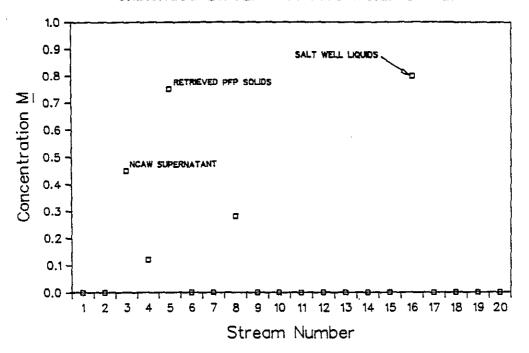
Even though the data used for the calculations presented in this appendix were taken from a single waste management scenario, the results presented here are applicable to almost any other credible scenario. An examination of the assumptions used for the calculations shows that a variety of situations are represented. Examples of this variety are the assumptions for the startup and shutdown of each major Hanford Site facility. During the period of this study, each facility is shut down before the year 2015. This means that the results of this study also reflect waste character when these plants are down. The only way that the results from this study would be invalid is if new process (not considered in these calculations) were to emit unusual chemicals in large quantities. An example of such a process would be the retrieval and processing of single-shell tank solids.

3A.4 CONCLUSION

The good correlation between laboratory analyses and waste origins in current inventory confirms that typical DST waste is a well blended mix of many different waste streams.

The good correlation between calculations of future DST waste composition and recent DST waste analyses suggests that the wide variations in individual waste stream components average after mixing. This eliminates any influence of waste streams that may vary from day to day. It appears that there has been no overall trend in the major constituents of dilute noncomplexed wastes that would set future wastes apart from what is already in inventory. The good correlation suggests that the data presented in Tables 3-1, 3-2, and 3-3 in Chapter 3.0 are useful for determining compositional ranges for future grout feed.

Grout Feed Concentration for AlO_2^{-} Unblended Streams at Five Molar Sodium



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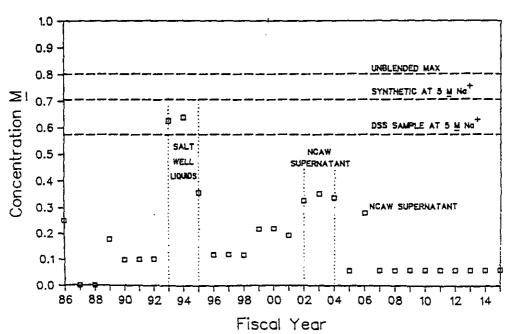
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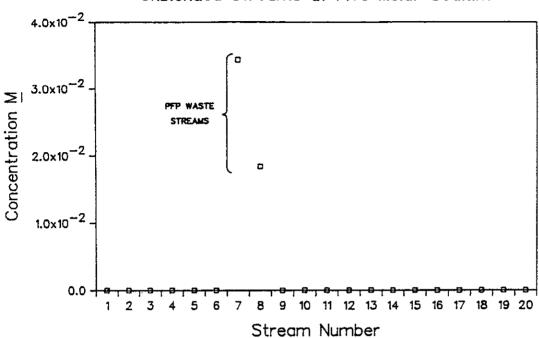
Grout Feed Concentration for AIO₂

Blended Streams at Five Molar Sodium



Grout Feed Concentration for Ca⁺³

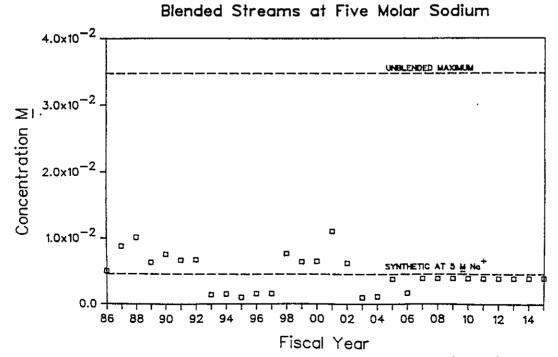
Unblended Streams at Five Molar Sodium



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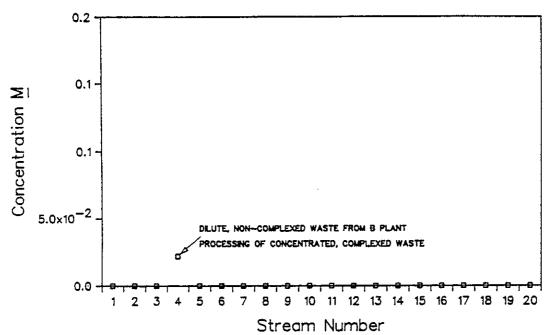
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Grout Feed Concentration for Ca⁺³



DSS AT 5 M No +: 18x10 -4

Grout Feed Concentration for CI Unblended Streams at Five Molar Sodium



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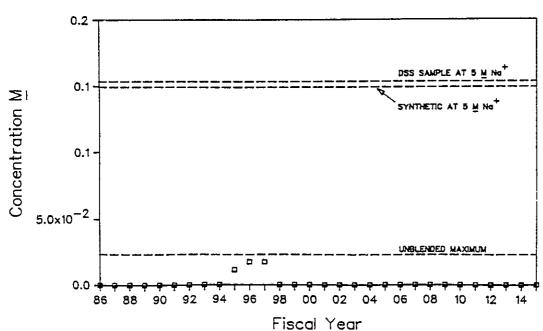
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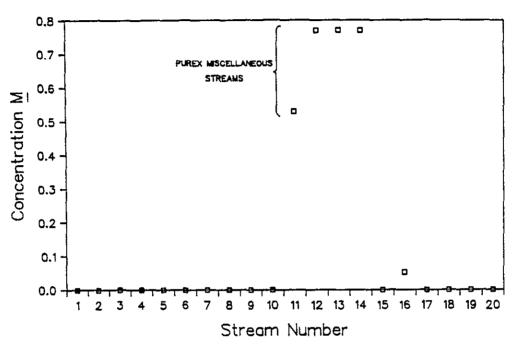
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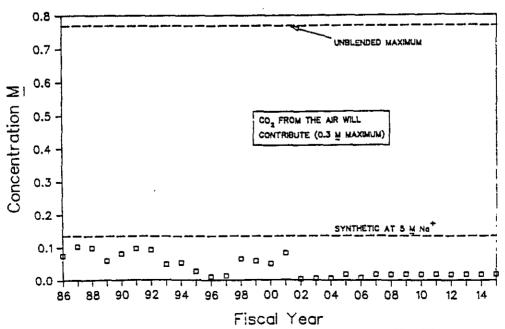
Grout Feed Concentration for CI⁻ Blended Streams at Five Molar Sodium



Grout Feed Concentration for ${\rm CO_3}^{-2}$ Unblended Streams at Five Molar Sodium



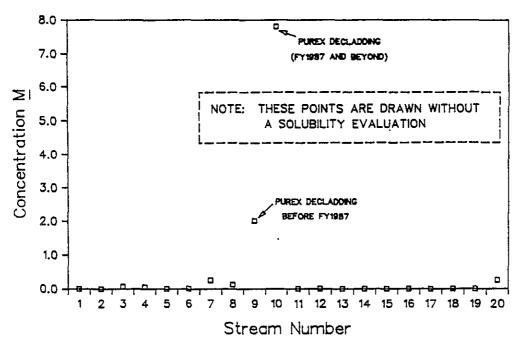
Grout Feed Concentration for ${\rm CO_3}^{-2}$ Blended Streams at Five Molar Sodium



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Grout Feed Concentration for F

Unblended Streams at Five Molar Sodium



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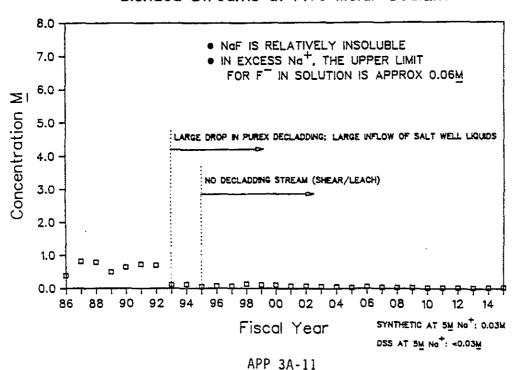
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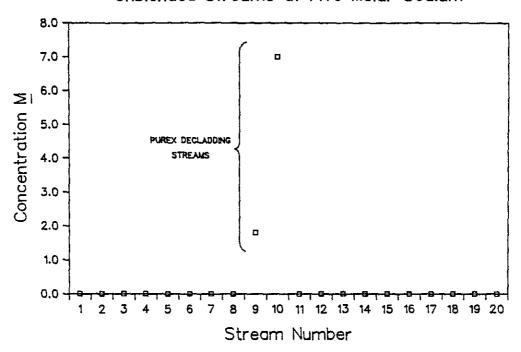
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Grout Feed Concentration for F

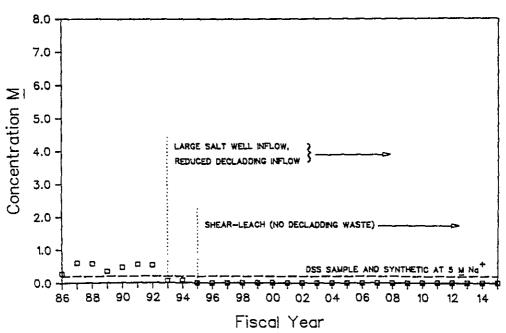
Blended Streams at Five Molar Sodium



Grout Feed Concentration for K^+ Unblended Streams at Five Molar Sodium

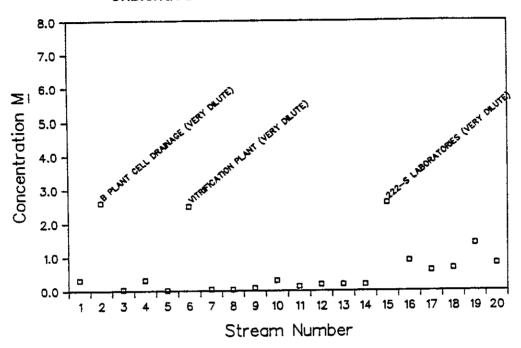


Grout Feed Concentration for K⁺
Blended Streams at Five Molar Sodium



APP 3A-12

Grout Feed Concentration for NO₂
Unblended Streams at Five Molar Sodium



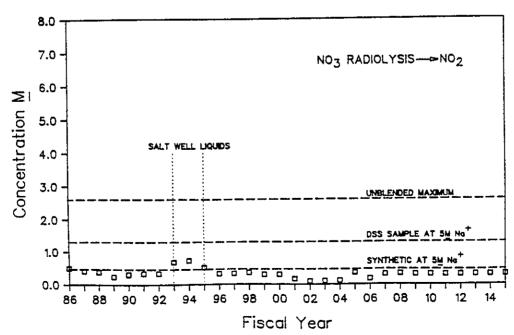
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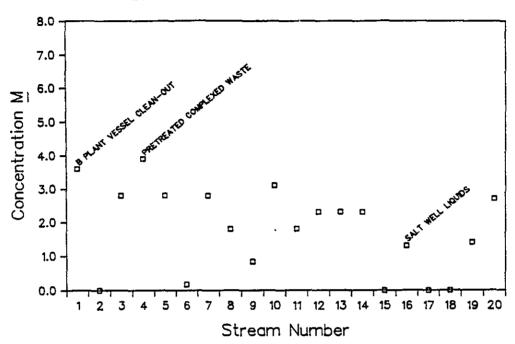
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Grout Feed Concentration for NO_2^{-} Blended Streams at Five Molar Sodium

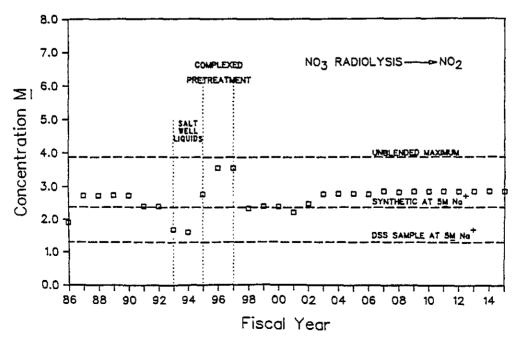


APP 3A-15

Grout Feed Concentration for NO₃
Unblended Streams at Five Molar Sodium

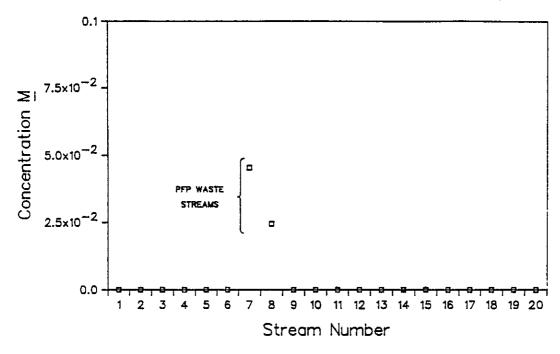


Grout Feed Concentration for NO_3^- Blended Streams at Five Molar Sodium



APP 3A-16

Grout Feed Concentration for Mg⁺²
Unblended Streams at Five Molar Sodium



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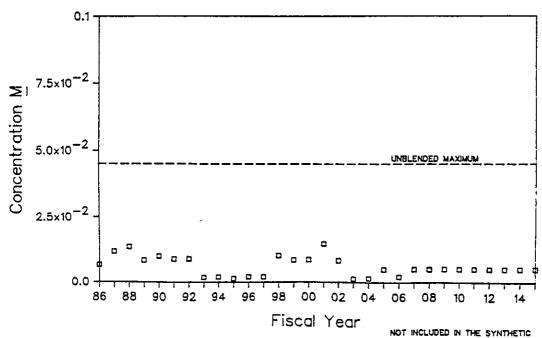
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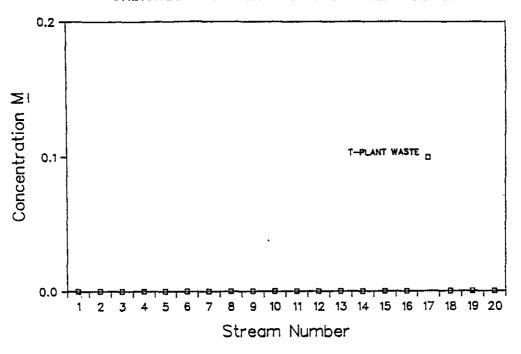
Grout Feed Concentration for Mg⁺²
Blended Streams at Five Molar Sodium



DSS SAMPLE AT 5 M Na : 43x10

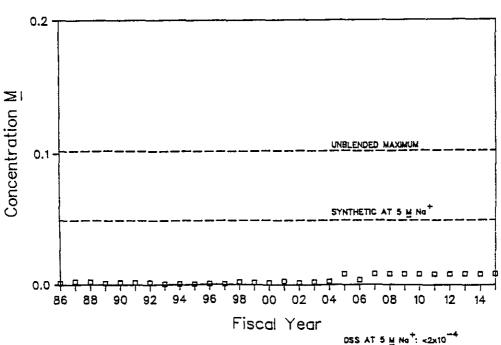
APP 3A-13

Grout Feed Concentration for MnO₄⁻ Unblended Streams at Five Molar Sodium



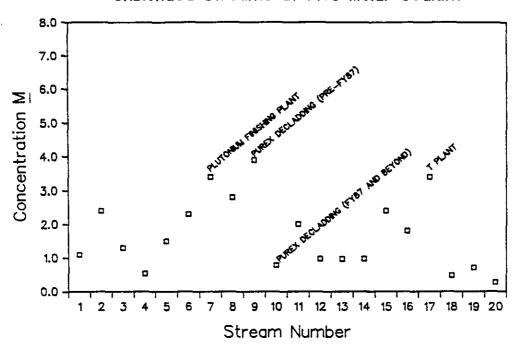
Grout Feed Concentration for MnO₄

Blended Streams at Five Molar Sodium

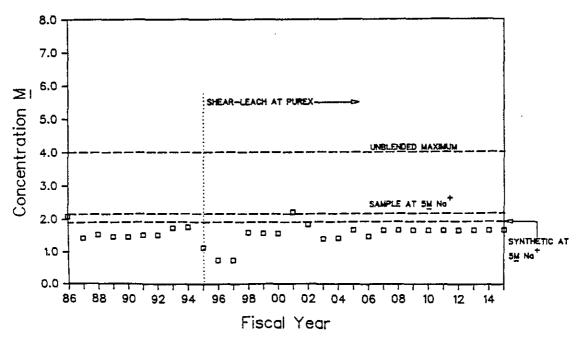


APP 3A-14

Grout Feed Concentration for OH⁻ Unblended Streams at Five Molar Sodium



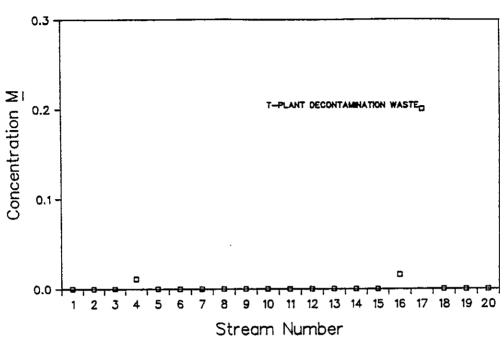
Grout Feed Concentration for OH Blended Streams at Five Molar Sodium



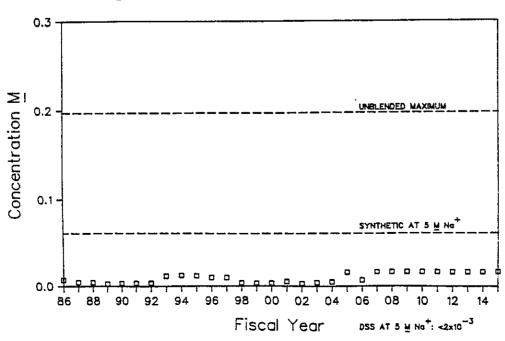
APP 3A-17

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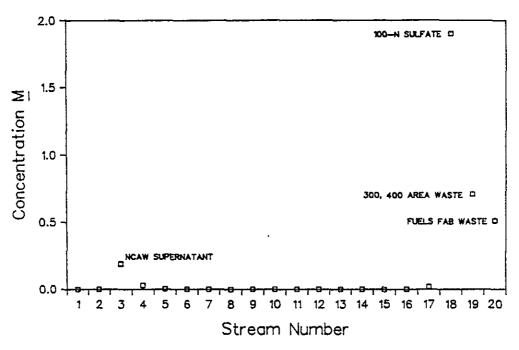
Grout Feed Concentration for PO₄⁻³
Unblended Streams at Five Molar Sodium



Grout Feed Concentration for PO₄⁻³
Blended Streams at Five Molar Sodium



Grout Feed Concentration for ${{\rm SO_4}^{-2}}$ Unblended Streams at Five Molar Sodium



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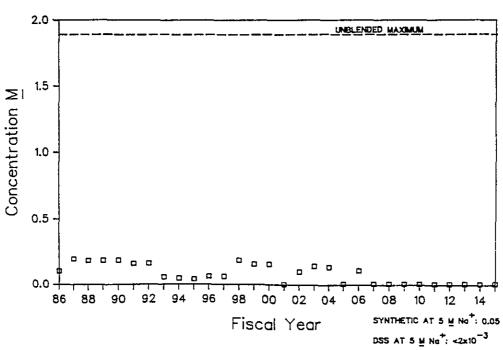
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Grout Feed Concentration for ${\rm SO_4}^{-2}$ Blended Streams at Five Molar Sodium



APP 3A-19

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APPENDIX 3B

LABORATORY ANALYSIS REPORTS FOR DOUBLE-SHELL TANK WASTE STORED IN TANKS 241-AN-106, 241-AW-101, AND 241-AN-103

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APPENDIX 3B

LABORATORY ANALYSIS REPORTS FOR DOUBLE-SHELL TANK WASTE STORED IN TANKS 241-AN-106, 241-AW-101, AND 241-AN-103

Sampling and Analysis Techniques, Tank 241-AN-103

The quantitative data presented for tank 241-AN-103 (Tables 3B-1, -2, and -3) are based on the analysis of two composite samples from the tank. The first sample was taken using the weighted-bottle method of ASTM-E-300 (ASTM 1986). The second sample was taken using the double-shell tank (DST) core sampling equipment. The tank contains 937,000 gal of concentrated DST waste.

The first sample was made up of three sub-samples taken from different elevations within the tank. The elevations sampled were 2 ft below the liquid surface, the tank midpoint, and the bottom of the tank liquid contents. The second sample was prepared by taking an equal volume from each segment of the core sample. Each sample was homogenized to give the analytical sample. Each analytical sample was analyzed for radiochemical, inorganic, and organic constituents by Pacific Northwest Laboratory. Each analysis was performed using either a modified EPA-certified method or an EPA-comparable method.

The detection limits for the organic analysis were estimated based on the recovery of standards added to the waste matrix. The detection limit estimated for semivolatile organic materials was 2.6 E-04 mg/g and for hydrophilic organic materials as 6.0 E-05 mg/g.

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Table 3B-1. Radionuclide Concentrations--Tank 241-AN-103.

Radionuclide	Concentration (Ci/L)	Standard deviation (Ci/L) ^a
H-3	4.0 E-06	5.7 E-06
C-14	2.0 E-06	2.8 E-06
Co-60	3.8 E-05	6.4 E-07
Se-79	4.3 E-05	
Sr-90	1.3 E-02	1.6 E-02
Nb-94	7.1 E-06	7.3 E-06
Tc-99	1.7 E-04	8.8 E-05
Ru-106	6.8 E-05	
I-129	5.2 E-07	5.3 E-07
Cs-134	1.1 E-04	1.5 E-04
Cs-137	7.5 E-01	5.7 E-03
U-234	5.3 E-08	2.4 E-08
U-235	1.1 E-09	5.2 E-10
U-238_	1.4 E-08	6.4 E-10
Np-237	2.0 E-08	8.5 E-09
Pu-238	9.8 E-07	5.9 E-07
Pu-239/240	1.9 E-06	9.1 E-07
Am-241	2.3 E-06	1.5 E-06
Cm-244	4.2 E-07	4.9 E-08

^aBased on the analysis of 2 composite tank samples.

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Sampling and Analysis Techniques, Tank 241-AN-106

Tank 241-AN-106 was sampled using the techniques of ASTM-E-300, (ASTM 1986) by tank farm operations personnel. Concurrent with the sampling of the tank the sludge level was measured. The sludge level in the tank was reported as less than 4 in. at all sludge measurement points. This sludge level corresponds to less than the detection limit of the sludge measurement system and therefore represents zero. Tank samples (Tables 3B-4, -5, -6) were obtained at the following heights as measured from the tank bottom: 1.5, 12, 22, and 32.5 ft. The samples were shipped to the 222-S Analytical Laboratory in the 200 West Area.

APP 3B-5

Table 3B-4. Radionuclide Concentrations--Tank 241-AN-106.

Radionuclide	Concentration (Ci/L)	Standard deviation (Ci/L) ^a
H-3 C-14 Se-79 Sr-90 Tc-99 I-129 Cs-137 Pu-238 Pu-239/240 U-238 ^b Np-237 Am-241	7.0 E-06 6.5 E-07 5.2 E-07 4.4 E-03 8.5 E-05 2.0 E-07 2.7 E-01 2.1 E-07 4.4 E-07 4.8 E-09 1.6 E-07 1.5 E-06	5.8 E-07 4.5 E-07 7.4 E-05 2.3 E-01 1.6 E-07

Note: No other isotopes were detected by gamma energy analysis at concentrations greater than method detection limit (1 E-05 - 1 E-07 Ci/L depending on energy of emission). The additional isotopes detected in other tanks would have been detected by this analysis.

aWhere standard deviations are reported they are based

on measurements from 4 samples.

^bCalculated from measured uranium concentration of 1.2 E-02 mg/g based on the assumption that all uranium present is present as U-238.

2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 17 18 19 20 21 21 22 3 3 LO 30 31 32 33 34 35 36 37 38 39 40 42 43 44 45 46 47 48

Table 3B-2. Inorganic Chemical Concentrations -- Tank 241-AN-103.

Chemical	Concentration (mg/g)	Standard deviation (mg/g) ^a
Ag	<1.0 E-02	0.0 E+00
A1 As	3.6 E+01 <1.0 E-01	1.3 E+01 0.0 E+00
Ba	<1.0 E-01	0.0 E+00
Be	<1.0 E-02	0.0 E+00
Bi	<3.0 E-01	2.8 E-02
Ca	5.4 E-02	5.9 E-02
Cd	1.0 E-02	0.0 E+00 9.9 E-01
Cl CN(total)	6.0 E+00 2.1 E-02	1.4 E-03
CN(free)	2.5 E-05	
CO ₃	5.6 E-00	
Cr	5.3 E-01	1.1 E-01
Cu	7.5 E-03	3.5 E-03
F Fe	4.6 E-01 4.4 E-02	3.5 E-01 4.7 E-02
H ₂ 0	3.8 E+02	3.6 E+00
Hg	1.0 E-02	1.4 E-02
K	9.5 E+00	7.1 E-01
Mg	1.8 E-02	1.1 E-02
Mn Mo	1.8 E-02 5.5 E-02	1.1 E-02 7.1 E-03
Na (13. <u>M</u>)	2.1 E+02	3.3 E+01
Ni	<1.5 E-02	7.1 E-03
NO ₃	1.0 E+02	4.0 E+01
NO2 OH	8.6 E+01 6.1 E+01	1.6 E+01 0.0 E+00
Pb	4.5 E-02	4.9 E-02
PO ₄	5.8 E-01	4.6 E-01
Sb	<1.0 E-01	0.0 E+00
Se s:	<1.3 E-01	3.5 E-02
Si SO ₄	1.7 E-01 1.0 E+00	1.7 E-01 1.1 E+00
Ti	<1.0 E-02	0.0 E+00
TOC	4.6 E+00	1.9 E+00
U	7.7 E-02	3.3 E-02
V W	<1.0 E-02 1.3 E-01	0.0 E+00
w Zn	3.0 E-02	4.2 E-02 2.8 E-02
Žr	<1.5 E-02	7.1 E-03
Density(g/mL)	1.6 E+00	0.0 E+00

Note: < = detection limit of analytical method. a Based on the analysis of 2 composite tank samples.

Table 3B-3. Organic Chemical Concentrations--Tank 241-AN-103.

Chemical	Concentration (mg/g)	Standard deviation (mg/g) ^a
[(Tri-n-butyl)di-ol]phosphate	7.0 E-03	
2-chloromethyl, hydroxymethylbenzene 2-Hydroxymethylbenzoic acid 2-Methylbenzoic acid 2-Methyl,hydroxymethyl benzene 4-Chloromethyl-o-xylene Alkyl,hydroxymethyl benzene Butanedioic acid C3-Alkylbenzene Chloroethyl,2-hydroxymethyl,	7.7 E-03 1.7 E-02 1.1 E-02 2.2 E-01 4.1 E-03 1.1 E-03 2.6 E-01 2.0 E-01	
benzoic acid Citric acid Dioctylphthalate ED3A EDTA Ethanedioic acid Ethyl,2-methyl,	* 8.0 E-03 1.1 E-02 1.5 E-02 3.0 E-03 5.3 E-02 2.6 E+00	1.6 E-02 1.2 E-02
hydroxymethylbenzenes Ethylbenzaldehyde Ethylxylene Heptadecanoic acid Heptanedioic acid Hexanedoic acid Hexanoic acid MAIDA Methylbenzaldehyde Methyltoluidine MICEDA n-C22H46 - n-C34H70	2.9 E-02 4.3 E-01 2.0 E-04 1.5 E-03 1.7 E-02 4.0 E-02 2.7 E-02 3.6 E-01 4.3 E-01 2.2 E-03 1.9 E-02 9.3 E-03	2.1 E-03
n-Dimethyltoluidine n-Dodecane n-Pentadecane n-Tetradecane n-Tridecane n-Undecane NTA Pentadecanoic acid Pentanedioic acid Propylbenzene Tri-n-butyl phosphate Trimethylbenzene Unknown phthalates	7.2 E-03 2.5 E-03 2.3 E-03 5.6 E-03 9.1 E-03 3.6 E-04 2.9 E-03 2.2 E-02 4.4 E-02 1.1 E-03 1.1 E-02 4.9 E-02 1.3 E-02	4.1 E-03 3.1 E-02 2.1 E-03

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Table 3B-5. Inorganic Chemical Constituents--Tank 241-AN-106.

Chemical	Concentration (mg/g)	Standard deviation ^a (mg/g)
Ag	<2.0 E-03	
Al	8.8 E+00	8.8 E+00
As	<2.2 E-03 1.4 E-02	8.2 E-03
B Ba	<3.2 E-03	0.Z L-03
Bi	<8.7 E-02	
Ca	5.6 E-02	4.3 E-02
Çq	<2.4 E-02	
Ce	<3.7 E-02	2.1 E+00
C1 CN	2.4 E+00 1.1 E-02	2.1 6400
CO ₃	1.9 E+01	
Cr	5.1 E-01	4.3 E-01
Cu	1.5 E-03	9.3 E -04
F	2.8 E-02	• -
Fe Han	7.8 E-03 6.7 E+02	
H ₂ 0 K	1.0 E-06	7.8 E-07
La	<3.1 E-04	
Li	<5.8 E-03	- •
Mg Mn	<9.9 E-04 <1.2 E-03	
Mo	2.7 E-02	1.6 E-02
Na (5.3 <u>M</u>)	1.0 E+02	5.1 E+ 0 1
Nd	<1.3 E-02	
Ni No-	4.4 E-02 2.8 E+01	6.2 E-03 2.3 E+01
NO ₂ NO ₃	6.6 E+01	5.5 E+01
OH	1.0 E+01	8.4 E+00
Р	4.1 E+00	4.1 E+00
Pb	<1.4 E-01	
Pd	<2.8 E-02 1.2 E+01	1.3 E+01
P0 ₄ Se	<4.5 E-04	1,3 ETV1
Si	2.3 E-02	1.2 E-02
S0 ₄	2.4 E+00	1.6 E+00
Ta	<1.3 E-01	
Ti TOC	<2.6 E-03 4.4 E+00	· • •
U	1.2 E-02	
Zn	<7.0 E-03	
Zr	<8.7 E-02	
Density(g/ml)	1.2 E+00	1.3 E-01

Note: < = detection limit of analytical method. aWhere standard deviations are reported they are based on measurements from 4 samples. S

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Table 3B-6. Organic Constituents--Tank 241-AN-106.

Organic compound	Molecular formula	Esti <u>concent</u> (<u>M</u>)	mated ration ^a (mg/g)
Citric acid	C ₆ H ₈ O ₇	2.7 E-02	4.3
Ethylenediamine- tetraacetic acid (EDTA)	C ₁₀ H ₁₆ N ₂ O ₈	4.0 E-03	1.0
Hydroxyacetic acid	C ₂ H ₄ O ₃	3.8 E-02	2.4
N-Hydroxyethylene- diaminetriacetic ac (HEDTA)	id C ₁₀ H ₁₈ N ₂ O ₇	1.7 E-02	3.9

^aEstimated from the measurement of total organic content and the organics listed in process flowsheets.

Sampling and Analysis Techniques, Tank 241-AW-101

Tank 241-AW-101 was sampled while the waste was being generated by the evaporation of dilute waste material. The DST waste product was sampled approximately every 24 h during the transfer to tank 241-AW-101. The samples were taken using an in-line sampler installed at the 242-A Evaporator. Four waste tank samples were taken for analysis. The tank now contains 1,037,000 gal of liquid DST waste and 84,000 gal of solids. The solids in the tank are a result of precipitation of materials from the waste as it cools and are thus included in the samples used for characterization.

The four individual samples were analyzed at the 222-S Analytical Laboratory in the 200 West Area for inorganic and radiochemical constituents. The four analytical results from the analysis of the individual samples were averaged to give two estimates of the tank composition. In addition to the analyses prepared by the 222-S Analytical Laboratory, the four samples were used to prepare two equal volume composites for analysis by Pacific Northwest Laboratory. The two composite samples were analyzed by Pacific Northwest Laboratory for radiochemical, inorganic, and organic constituents. The analysis by Pacific Northwest Laboratory resulted in two additional estimates of the tank composition, resulting in a total of as many as four estimates of tank composition (Tables 3B-7, -8, and -9).

The detection limits for the organic analysis were estimated based on the recovery of standards added to the waste matrix. The detection limit estimated for semivolatile organic materials was 3.2 E-04 mg/g and for hydrophilic organic materials as 5.0 E-05 mg/g.

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Table 3B-7. Radionuclide Concentrations--Tank 241-AW-101.

Radionuclide	Concentration ^a (Ci/L)	Sample standard deviation ^a (Ci/L)
H-3 C-14 Co-60 Se-79b Sr-90 Nb-94 Tc-99 Ru-106 I-129 Cs-134 Cs-137 U-234 U-235 U-238 Np-237 Pu-238 Pu-239/240 Am-241 Cm-244	1.9 E-05 1.5 E-06 2.7 E-05 4.9 E-07 1.5 E-02 4.2 E-05 1.1 E-04 2.0 E-02 1.3 E-07 5.6 E-03 5.0 E-01 2.0 E-08 2.5 E-09 2.1 E-08 9.4 E-09 1.0 E-06 2.2 E-06 2.7 E-06 6.6 E-08	1.1 E-05 1.2 E-06* 2.8 E-06 5.7 E-07* 9.5 E-03* 4.2 E-06 1.3 E-04* 2.0 E-02* 8.1 E-08 3.3 E-03* 2.0 E-01* 2.2 E-08 1.6 E-09 9.9 E-09 8.5 E-10 5.7 E-07 1.2 E-06* 2.3 E-06* 4.3 E-08

^aReported values based on average of two samples except where indicated with an asterisk where four samples were used. ^bEstimated value based on Tc-99 concentration.

Table 3B-8. Chemical Concentrations--Tank 241-AW-101.

Chemical	Concentration (mg/g)	Sample standard deviation ^a (mg/g)
Ag Al As Ba Be Bi Ca Cd Cl CN CO3 Cr Fe H20 Hg K Mg Mn Mo Na No3 NO2 OH Pb PO4 Sb Se Si SO4		
Ti TOC U V W Zn Zr	7.5 E-01 6.1 E-02 <1.0 E-02 <1.0 E-01 <1.0 E-02 <1.0 E-02	7.1 E-02 2.0 E-02
Density(g/ml)	1.5 E+00	1.4 E-02

Note: < = detection limit of analytical method.

aWhere a sample standard deviation is reported it is based on the mean of four measurements.

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Concentration (mg/g)	Sample standard deviation ^a (mg/g)
3.5 E-02 4.4 E-03 1.2 E-03 6.3 E-04 1.2 E-02 7.2 E-03 5.5 E-04 4.7 E-03 1.3 E-02 1.1 E-03 5.3 E-04 4.9 E-03 9.5 E-03 2.2 E-03 5.0 E-03 2.7 E-04 1.8 E-02	7.4 E-03 5.6 E-03 1.5 E-03 4.9 E-05 1.8 E-03 1.4 E-05 7.4 E-04 1.2 E-02 4.9 E-04 4.3 E-03 2.0 E-03 1.4 E-04 1.3 E-04 2.5 E-02
	(mg/g) 3.5 E-02 4.4 E-03 1.2 E-03 6.3 E-04 1.2 E-02 7.2 E-03 5.5 E-04 4.7 E-03 1.3 E-02 1.1 E-03 5.3 E-04 4.9 E-03 9.5 E-03 2.2 E-03 5.0 E-03

 $^{\rm a}{\rm Where}$ a sample standard deviation is reported it is based on the mean of two measurements.

REFERENCE

ASTM, 1986, Recommended Practice for Sampling Industrial Chemicals, ASTM-E-300, American Society for Testing and Materials, Philadelphia, Pennyslvania.

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APPENDIX 3C

THERMAL ANALYSIS IN SUPPORT OF GROUT TEMPERATURE LIMITS AND HEAT-LOADING GUIDELINES

APPENDIX 3C

THERMAL ANALYSIS IN SUPPORT OF GROUT TEMPERATURE LIMITS AND HEAT-LOADING GUIDELINES

The information in this appendix identifies the principles related to grout curing at varying temperatures. The tests were conducted on a nondangerous waste form. The temperature/curing relationship identified also should be valid for grouted dangerous wastes.

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APPENDIX 3C

THERMAL ANALYSIS IN SUPPORT OF GROUT TEMPERATURE LIMITS AND HEAT-LOADING GUIDELINES

This appendix presents the results of laboratory investigations that were performed to identify a temperature range for grout curing, within which grout will retain properties that make it desirable as a waste disposal medium.

3C.1 INITIAL TEMPERATURE TESTS AND RESULTING LIMITS

Grout samples were prepared for testing using a simulated phosphate/sulfate waste (PSW) solution. These samples were subjected to various temperatures during mixing and curing processes. The results of these tests are summarized below.

- The freezing of grout at -7 °C resulted in the formation of ice crystals that caused the grout form to expand and increase in porosity.
- Grouts cured at 0 °C and 5 °C did not harden at an acceptable rate.
- Curing temperatures of between 10 and 100 °C resulted in grouts that had acceptable physical properties with no signs of cracking or increased porosity.
- Grout cured at 105 °C severely cracked and dried even though 100% relative humidity was maintained.

Based on these tests, a lower limit of 10 °C and an upper limit of 100 °C were identified. A temperature of 90 °C was chosen as the upper temperature limit for this study to provide a 10 °C margin of safety. Because the average temperature of the soil at the Hanford Site (below the freeze level) is 13 °C, it is very unlikely that the temperature of grout will fall below 10 °C. Therefore, the lower temperature standard for double-shell tank (DST) grout was set at 10 °C.

3C.2 THERMAL ANALYSIS USING COMPUTER MODEL

Heating generation within a grout monolith that could cause the temperature to exceed the limit could come from three sources: heat from chemical hydration reactions, other exothermic chemical reactions (e.g., between waste and grout-forming materials), and heat from radionuclide decay. The amount of heat generated from hydration reactions is determined by the concentration of cement in the grout. This cement concentration is determined solely by the final physical properties that are desired in the grout. Therefore, the grout temperature must be controlled by a variable other than cement concentration (i.e., by limiting the heat generated by

other than cement concentration (i.e., by limiting the heat generated by radionuclide decay or chemical reactions, or by installing a cooling system or other modification to the disposal system). Thus, the concentrations of heat-producing radionuclides and reactive chemicals in the grout must be limited or the disposal system must be altered.

To determine these preliminary radionuclide concentration guidelines, a transient thermal analysis was performed on a portion of a theoretical grout monolith in a grout disposal field. The computer code TEMPEST (Trent et al. 1983) was the preliminary analytical tool used to perform the analysis. A two-dimensional TEMPEST model was used to find the time-versus-temperature relationship of a grout monolith containing a given loading (concentration) of a particular radionuclide. The analysis also accounted for heat produced via the cement hydration reactions. The hydration heat was determined by the following equation developed for the Hanford Site grout technology program:

$$H = 3.86 \qquad 0 < t \le 24$$

$$H = -0.1 + 4.04 \times 10^{-4} t + 2 \times 10^{3} t^{-2} \qquad 24 < t \le 168$$

$$H = \frac{(459 - 47.7 \ln t) [0.1525 (\ln t) - 7.92 \times 10^{-3} (\ln t)^{2}]}{34.64 t} \qquad 168 < t \le 8760$$

$$H = 0 \qquad t > 8760$$

where

H = heat generation per pound of cement (Btu/lb·h).t = time (h).

A three-dimensional TEMPEST analysis could be performed to eliminate some of the conservatism that resulted from two-dimensional modeling of the grout system.

3C.2.1 Modeling Assumptions and Parameters

The monolith section (Figure 3C.1) was modeled using the following physical dimensions. Grout is poured into a concrete vault that is 125 ft long, 50 ft wide, and 34 ft deep (inside dimensions). The vault has 3.5-ft-thick walls, a 2.5-ft-thick bottom, and a 2-ft 2-in.-thick top. A liner covers the inside surface of the vault, and consists of the following components (from the inside out): 60-mil high-density polyethylene (HDPE), 1/4-in. geotextile, and 1/4-in. drainage net. The vaults are built to contain a pair of monoliths 34 ft apart (27 ft between the outside edges of the vault walls). The space between the pair of monoliths is filled with soil. Figure 3C-2 is a schematic of a monolith in a vault, showing the various material parts as assumed for the computer model.

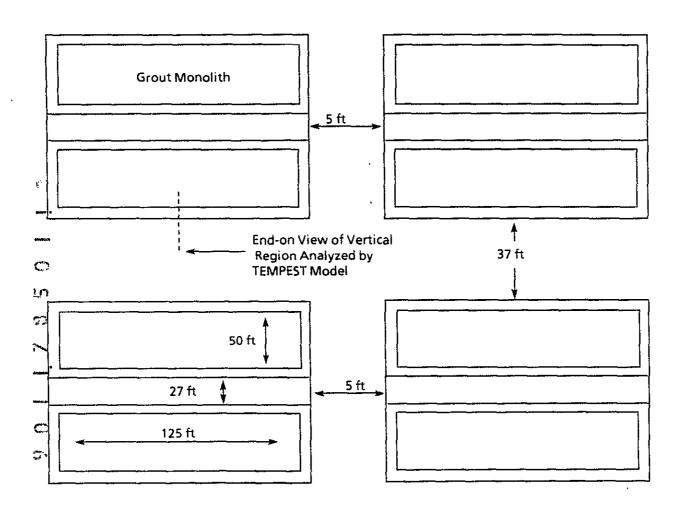


Figure 3C-1. Sections of the Grout Monolith and Vault that were Modeled.

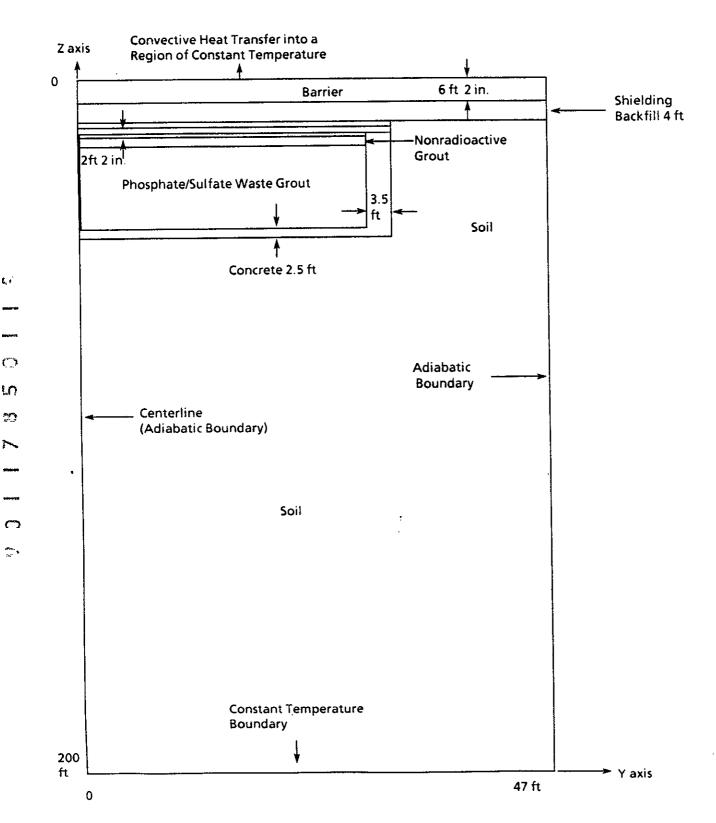


Figure 3C-2. Schematic of the Grout Monolith and Vault Model.

The grout monoliths were modeled as being poured in two distinct steps. First, radioactive grout is poured into the vault until the vault is within 4 ft of being full. As modeled, the grout cures in this configuration for 1 yr after the initial pour. After 1 yr, a nonradioactive grout is poured into the vault to fill the 4-ft head space. Also at 1 yr after the initial pour, a 4-ft-thick shielding backfill of soil and a 6-ft 2-in. soil barrier are placed over the vault.

The top boundary of the model was assumed to be at a constant ambient temperature of 15 °C and thermally connected to the model by an overall heat transfer coefficient of 2 Btu/h·ft 2 °F. The bottom boundary was assumed to be 200 ft below grade and at a constant temperature of 13 °C. Lines of symmetry between the different vaults in a disposal field were treated as adiabatic boundaries.

The following is a list of other assumptions and parameters used during the temperature analyses:

- Initial grout temperature: 27 °C
- The grout contained 1.8 lb of cement/gal of grout (7.5 lb of dry grouting solids/gal of grout)
- Grout density: 83 lb/ft³
- Thermal conductivity of the grout: 0.4 Btu/h·ft°F
- Specific heat of the grout: 0.66 Btu/lb°F
- Thermal conductivity of HDPE: 0.19 Btu/h·ft°F (Perry and Chilton 1973)
- Thermal conductivity of the geotextile: 0.027 Btu/h·ft°F (Kreith 1976)
- Thermal resistance of the drainage net was neglected
- Thermal capacitance of the liner was neglected; only its thermal resistance was accounted for
- Soil properties: thermal conductivity 0.25 Btu/h·ft°F, density - 113 lb/ft³, and specific heat - 0.22 Btu/lb°F
- Concrete properties: thermal conductivity 0.54 Btu/h·ft°F, density - 144 lb/ft³, and specific heat - 0.21 Btu/lb°F
- The vault's concrete lid contains internal hollow pipes; an effective resistance of 0.17 Btu/h·ft°F was calculated for this structure
- Only one of seven nuclides was assumed present in the grout for each time the computer code was run

 Concentration of each radionuclide was assumed constant throughout the grout monolith.

3C.2.2 Development of Heat-Loading Guidelines

Using the computer model, the loading guidelines for seven different radionuclides (Table 3C.1) were determined. As mentioned, the analyses were performed assuming that only one radionuclide was present in the grout for each of the computer runs. Although the TEMPEST model did not directly account for decay heat from daughter products, the heat-generation rates used did account for the decay heat from important daughter products (e.g., 90 Y for 90 Sr). Table 3C-1 shows the radionuclides investigated, their half-lives, and the rates at which they generate heat.

Table 3C-1. Heat Generation Rates and Half-Lives for the Radionuclides Studied.

Antimony-125-tellurium-125m Cobalt-60 Strontium-yttrium-90	Half-life (yr)	Heat generation rate (W/g)
Cerium-praseodymium-144	0.8	25.6
	1.01	33.1
Cesium-134	2.05	13.8
Antimony-125-tellurium-125m	2.7	3.5
Cobalt-60	5.26	17.7
Strontium-yttrium-90	28.1	0.93
Cesium-137-barium-137m	30.0	0.42

After Benedict et al. (1981); American Institute of Physics (1958).

For the TEMPEST calculations, an upper limit of 90 °C was used. The limit was assumed to have been exceeded when the temperatures at any location in the monolith surpassed approximately 90 °C. Usually, this location was at or near the center of the grout monolith.

The analyses revealed that the peak temperature of the grout was very sensitive to small changes in the radionuclide concentrations in the grout. For example, increasing the amount of 90 Sr from 1.6 g/m³ (230 Ci/m³) to 2.0 g/m³ (280 Ci/m³) caused the peak temperature to increase 18 °C. Because of this sensitivity and the computer time involved in a calculational run, when a radionuclide concentration was found that was close to 90 °C, this value was taken to be the guideline (i.e., the value that gave exactly 90 °C was not searched for). Table 3C-2 lists the peak temperatures that were caused by the radionuclides and the times at which these temperatures were reached. The corresponding maximum concentration guidelines for these radionuclides in grouted waste are listed in Section 3.4. Figure 3C-3 contains plots of the time-versus-temperature relationships in the DST grout for the seven radionuclides studied.

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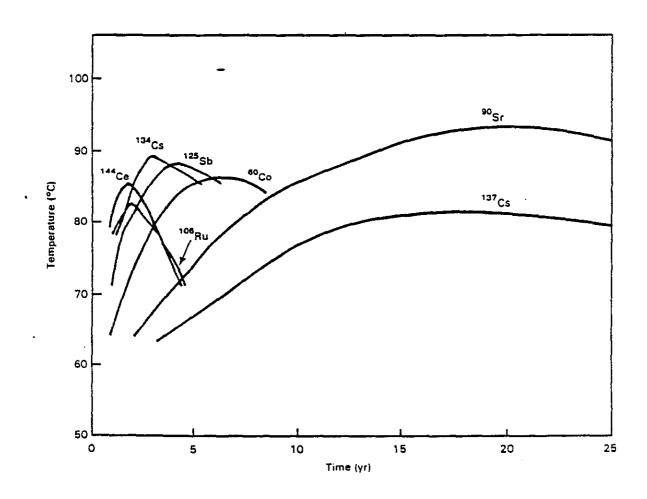
19 <equation-block> 20

Table 3C-2. Peak Temperatures and Times of Peak Temperatures for the Radionuclides Studied.

Radionuclide	Peak (°C)	Time of peak (yr after pour)
Cerium-praseodymium-144	86	1.8
Ruthenium-rhodium-106	84	2.0
Cesium-134	91	2.9
Antimony-125-tellurium-125m	90	4.0
Cobalt-60	88	6.4
Strontium-yttrium-90	94	19.0
Cesium-137-barium-137m	83	19.0

17 c. 3C.3 METHODS OF DETERMINING COMPLIANCE WITH TEMPERATURE LIMITS

Because all seven of these radionuclides will probably be present in an 21 actual grout monolith, a method to combine the seven individual concentration 22 guidelines into one guidance limit is desired. One method is to perform a sum-of-the-fractions analysis on the actual inventory, using the individual 4th guidelines. This method is very conservative because it assumes that all the radionuclides cause the temperature to peak at the same time, which is actually not true (see Figure 3C-3). A less conservative method would be to perform two different sum-of-the-fraction analyses for two different time periods: one for radionuclides that cause the temperature to peak before the 8 yr after the pour (i.e., all radionuclides except 137Cs and 90Sr) and one for radionuclides that cause the temperature to peak <u>after</u> the 8 yr after the pour (137 Cs and 90 Sr). This method combines only radionuclides that cause the temperature to peak at about the same time, which is less 33 conservative than assuming that they all occur at the same time.



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Figure 3C-1. Time-Versus-Temperature Relationship in a Grout Monolith for the Radionuclides Studied, at Temperature-Loading Guidelines.

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3C.4 REFERENCES

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- Benedict, M., T. H. Pigford, and H. W. Levi, 1981, *Nuclear Chemical Engineering*, Second Edition, McGraw-Hill Book Company, New York, New York.
- Kreith, F., 1976, *Principles of Heat Transfer*, 3rd Edition, IEP-A Dun-Donnelly Publisher, New York, New York.
- Perry, R. H. and C. H. Chilton, 1973, Chemical Engineers' Handbook, McGraw-Hill Book Company, New York, New York.
- 17 Trent, D. S., L. L. Eyler, and M. J. Budden, 1983, TEMPEST: A Three18 Dimensional Time-Dependent Computer Program for Hydrothermal
 19 Analysis, I: Numerical Methods and Input Instructions, PNL-4348, Pacific
 20 Northwest Laboratory, Richland, Washington.
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APPENDIX 3D

TEST RESULTS FOR EXTRACTION PROCEDURE TOXICITY TESTING

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APPENDIX 3D

TEST RESULTS FOR EXTRACTION PROCEDURE TOXICITY TESTING

The extraction procedure (EP) toxicity test was performed on an intact grout sample produced with actual waste from tank 241-AN-106. This test is U.S. Environmental Protection Agency (EPA) Test Method 1310 (EPA 1986), which is intended to determine whether a waste exhibits the characteristics of EP toxicity. A 100-g sample was placed in a 2-L container, along with distilled water equal to 16 times the sample weight. During the test the sample container was tumbled at 30 r/min, and periodically the pH was measured and adjusted to 5 ± 0.2 using a 0.5 N acetic acid solution. The pH adjustment continued for 6 h. After 24 h, the pH was measured and additional acid was added to reduce the pH to 5. Agitation continued for an additional 4 h. At the end of the test, distilled water was added to bring the total solution weight equal to 20 times the sample weight. An aliquot of the solution was then filtered through a 0.45-mm filter and submitted for ICP analysis for specific metals. The mercury level of the leachate was determined using a flameless atomic absorption method.

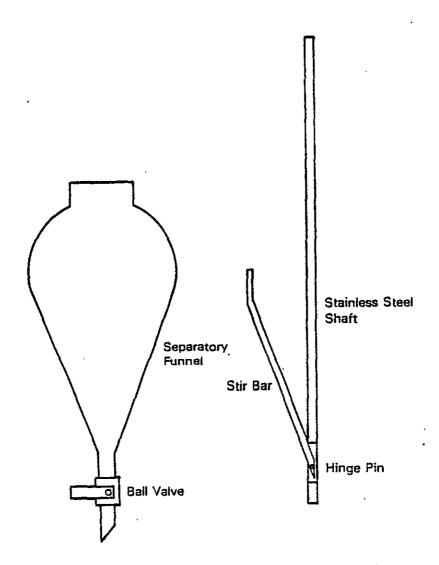
3D.1 TANK 241-AN-106 LIQUID WASTE HANDLING

Four lead-shielded pigs containing plastic bottles of liquid waste were obtained from Westinghouse Hanford Company. To create a composite waste sample, a beaker was placed on a stirrer/hot plate and the contents of one pig's bottle were poured into the beaker. Visual inspection showed a yellowish, clear fluid with no suspended solids. No residual precipitate was left in the bottle. Once the solution in the beaker reached >35 °C, a second pig was opened and its bottle's contents were added to the beaker. The procedure was repeated for the remaining bottles. At no time did the mixture show signs of precipitation or cloudiness. Because the liquid waste in each pig's bottle was radioactive, contents were added without measuring volumes. However, conversations with Westinghouse Hanford Company personnel indicated that each bottle contained about the same volume (i.e., 60 ± 10 mL).

3D.2 GROUT SAMPLE PRODUCTION

Grout from tank 241-AN-106 was prepared by mixing 1,080 g of dry blend with 1 L of liquid waste (9 lb/gal). The dry blend is a mixture of 47 wt% ground blast-furnace slag; 47 wt% class F fly ash from Centralia, Washington, and 6 wt% type I/II portland cement.

A small-volume mixing apparatus was built using a plastic 250-mL separatory funnel as the mixing chamber. A schematic of the apparatus is shown in Figure 3D-1. A stainless steel ball valve replaces the original stopcock at the bottom of the funnel and also allows more effective grout discharge. A paddle was constructed of a 1/4-in. stainless steel shaft and a metal shaft that is attached to the shaft with a hinge pin. The pin allows the bar to fold to the shaft for insertion into the funnel. The bottom



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Figure 3D-1. System Used to Mix Small Batches of Radioactive Grout.

portion of the shaft fit into the opening of the ball valve to minimize 'dead space' and to prevent wobbling during mixing. The paddle was rotated with a variable speed motor. During mixing, the bar swings out to the walls of the funnel. The mixing shaft extends above the separatory funnel and through a powder funnel used to add grout solids.

After mixing, the grouts were placed in polyethylene vials with tight-fitting lids and cured in an oven at 42 ± 2 °C. A 65-mL specimen was prepared on October 28, 1987. On December 7, 1987, the sample was removed from the oven and further cured at room temperature until February 17, 1988, when the EP toxicity test was performed.

3D.3 RESULTS OF EXTRACTION PROCEDURE TOXICITY TEST

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The results of the analysis of filtered leachate from the EP toxicity test are shown in Table 3D-1. In most cases, the metal concentrations are below the analytical detection limit and all the observed concentrations are below the regulatory limits. Thus, based on testing one sample, it appears that grout made from the waste in tank 241-AN-106 is not toxic per the EP toxicity test protocol.

Table 3D-1. Extraction Procedure Toxicity Results of Radioactive Grout in Tank 241-AN-106.

<u>Element</u>	Analyzed concentration (mg/L)	Regulatory limit (mg/L)
Ag	<0.01	5
As	<0.25	5
Ba	0.48	100
Cd	<0.01	1
Cr	0.07	5
Hg	0.0001	0.2
РĎ	<0.10	5
Se	<0.25	1

3D.4 REFERENCE

EPA, 1986, Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. Environmental Protection Agency, Washington, D.C.

APP 3D-3

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APPENDIX 3E

TEST RESULTS FOR TOXICITY TESTING OF DOUBLE-SHELL TANK GROUT

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APPENDIX 3E

TEST RESULTS FOR TOXICITY TESTING OF DOUBLE-SHELL TANK GROUT

The classification of double-shell tank (DST) material has been determined both by the book designation methods of WAC 173-303 (Ecology 1989). and by toxicology testing using methods prescribed by the Washington State Department of Ecology (Ecology), WDOE 80-12 (Ecology 1981). The results of aquatic toxicity testing showed that the DST waste material is an extremely hazardous waste (EHW) based on the criteria of WAC 173-303 (Ecology 1989). The testing in rats indicated that the DST waste was only a dangerous waste (DW). Based on these results, the waste material was classified as an EHW for toxicity.

Samples of grout prepared from the waste were also tested for toxicology 19 12 using the fish and rat bioassay procedures of WDOE 80-12. The results of the aquatic toxicology test showed that the grouted waste material was significantly less toxic than the waste material itself. One of the candidate grout formulations tested was a DW, and the other was an undesignated waste based on the criteria of WDOE 80-12. The rat toxicology testing on grouts made from the DST waste material verified these classifications.

3E.1 MATERIALS USED IN TOXICOLOGY TESTING

The high radiation levels associated with the DST waste material make testing of the waste or grouts made from the waste prohibitive in terms of radiation exposure to personnel. Therefore, all formulation development testing and verification is completed using a nonradioactive compositionally representative (NRCR) sample of the waste. The NRCR waste sample is an accurate representation of the chemical constituents present in the waste, prepared from detailed analysis of the waste streams for their chemical constituents and knowledge of the processes that generate the wastes.

The chemical analysis of the waste samples are used to develop a procedure for preparation of the NRCR waste. This procedure is designed to simulate the generation process of the waste and result in a material that is both compositionally and rheologically representative of the waste. The composition of a NRCR waste sample for the general DST waste class is presented in Table 3E-1. All testing was conducted using samples of this NRCR waste and grouts made from this waste material.

The two dry-material formulations were tested for toxicology; the formulations are identified as DS-3, and DS-19. These formulations are two of several candidate grout formulations currently under the process of consideration for the immobilization of the DST waste material. The dry materials used in these formulations and their relative proportions are shown in Table 3E-2. It must be remembered that these formulations are only two of many dry-material formulations being considered for the immobilization of this

.

Table 3E-1. Concentrations of Individual Components in the Nonradioactive Compositionally Representative Double-Shell Tank Waste.

<u>Specie</u>	mol/L
Ag	3.00 E-03
Al	1.50
As	8.16 E-07
Ba	9.08 E-03
Cl	2.18 E-01
Ca	1.00 E-02
Cd	1.40 E-04
CuII	2.00 E-04
SO4	1.05 E-01
Fe (ferric)	5.04 E-02
PO4	1.19 E-01
Hg	2.93 E-05
K	4.97 E-01
OH	4.1
F	5.91 E-02
Mn	1.00 E-01
Mo	1.01 E-03
Na	10.6
B4 ^O 7	4.85 E-03
CO ₃	2.99 E-01
Cr2 ^O 7	2.22 E-02
NO3	4.98
NO2	9.99 E-01
Ni	9.99 E-04
Pb	2.47 E-05
Se	1.10 E-04
Zn	4.97 E-02

waste and do not necessarily represent the final formulation that will be used for the processing of this waste.

3E.2 TOXICOLOGY TESTING CONDUCTED

Two types of materials were tested for their toxic hazard as measured by the test methods specified in WDOE 80-12 (Ecology 1981):

- The DST waste material
- The grouted-waste material.

The results of this testing are summarized in Table 3E-3, and the test reports are attached.

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36 \bigcirc Table 3E-2. Dry-Blend Compositions--Candidate Dry-Material Formulations.

DS-3	DS-19
	<u>(wt%)</u>
30	0
64	47.5
6	0
0	47.5
0	5
=====	=====
100	100
7	8
	(wt%) 30 64 6 0 0

Two major conclusions can be drawn from the data presented in Table 3E-3.

- The DST waste material is classified as an EHW, because 100% mortality of the fish was observed at the 100 p/m test level.
- The waste grouted using formulation DS-3 is classified as an undesignated waste by aquatic and rat toxicity testing. The testing of the material at the 0.5 g/kg (DW/EHW) level is not required for waste classification because the grouted waste cannot test worse than the untreated waste; the dry materials used in treating the waste are recognized as nontoxic materials. These test results classify DST waste solidified with the DS-3 formulation as an undesignated waste by the criteria of WDOE 80-12 (Ecology 1981).

The waste grouted using formulation DS-19 is classified as a DW by aquatic toxicity testing. The rat testing of this material confirms this designation. Based on these test results, DST waste solidified using the DS-19 formulation is a DW by the criteria of WDOE 80-12 (Ecology 1981).

Table 3E-3. Toxicology Testing Results Summary.

Material tested	Concentration tested	Test species	Resulta	Classification
		Waste samples test	ed	
DST waste DST waste	100 p/m 1,000 p/m	Fish Fish	(30/30) (30/30)	EHW
DST waste DST waste	5 g/kg 0.5 g/Kg	Rat Rat	(10/10) (00/10)b	DW
	DS	-3 Grout Samples T	ested	
DST grout DST grout	1,000 p/m 100 p/m	Fish Fish	(01/30) (02/30)	Undesignated
DST grout DST grout	5 g/kg 0.5 g/kg	Rat Rat	(01/10) Not required	
	DS-	-19 Grout samples	tested	
DST grout DST grout	1,000 p/m 100 p/m	Fish Fish	(16/30) Not required ^C	DW
DST grout DST grout	5 g/kg 0.5 g/kg	Rat Rat	(05/10) Not required ^d	

Note: The fish used for all aquatic toxicity testing were rainbow trout. The specimens used for all mammalian toxicity testing were male sprague dawley albino rats.

bTesting not required for classification.

aNumber of fatalities/number of test specimens.

^CExpected result is no fatalities, because of low fatality rate at 1,000 p/m level. dGrouted waste cannot test worse than the raw waste.

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3E.3 REFERENCES

Ecology, 1981, *Biological Testing Methods*, WDOE 80-12, Washington State Department of Ecology, Olympia, Washington.

Ecology, 1989, *Dangerous Waste Regulations*, Washington Administrative Code WAC 173-303, Washington State Department of Ecology, Olympia, Washington.

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APP 3E-6

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ATTACHMENT TO APPENDIX 3E

TEST REPORTS FROM TOXICOLOGY TESTING

E.V.S. CONSULTANTS ACUTE LETHALITY BIOASSAY RECORD

Client- Hart Crawser E.V.S. Project 6- 2/23,1-03 Hork Order 8- 8/36494	E.V.S. Analyst(s)- <u>BC 5+ EC.</u>
Identification- Ox J. zer Amount Received- Date Collected- Date Received- DH- Dissolved Oxygen (mg/1)- Conductivity (umbos/cm)- J. Zer J. Zer A J.	Bloassay Type-96. In UNOE H W.T.T. Test Initiation Date- Dec 12/1996
Other- DILUTION AND CONTROL HEDIUM Fresh Mater (dechlorinated)- Salt Water (Burrard Inlet)- pH Dissolved Oxygen (mg/1)- Conductivity (umhos/cm)- Hardness (mg/1 as CaCO ₃)- Alkalinity (mg/1 as CaCO ₃)- Salinity (°/ _{oo})- Other-	TEST SPECIES Rainbow Trout- Threespine Stickleback- Daphnia (B. magna)- Amphipod (R. abronius)- Other-
TEST CONDITIONS Temperature (°C)- 12.0-13.C pH Range- 58-11.1 Dissolved Oxygen Range- 9.0-10.3 Conductivity Range- Aeration (7.5 cc/min./1)- 1671 Photoperiod (L:0-in hours)- 14:10 No. Fish/Test Volume- 10/20.1 Fish Loading Density (9/1)- 0.31	
Handas Wasto (1)	Solzo decitho @ 100 mg/2 (ppm

APP 3E-8

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SAMPLE Dr.lizer	ACUTE LETHALITY BIOASSAY DATA
DATE COLLECTED	

E.V.S. PROJECT NO. 2/231-63

WORK ORDER NO. 360494

	TEST DATE	NO.	myh		PERCENT SURVIVAL DISSOLVED OXYGEN TEMPERATURE (1 to 96 hours) (mg/L) (°C)																												pH ·				pH.			pH.				pH					CONDUCTIVITY (umhos/cm)			LK g/L	HARD mg/L	
LAB NO.	TIME	FISH/ VOL.	CONC.	,— 	2	4	8	18	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	96		0	96	0	96																				
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MEAN FISH LENGTH (min) 3% RANGE 12-43 MEAN FISH WEIGHT (g) 2 RANGE 0.36-092	DATA VERIFIED BY A. KULLDRULL E.V.S. CONSULTANTS



January 13, 1986

EVS Consultants 2335 Eastlake Avenue Seattle, WA 98102

ATTN: Carolyn Evans

SUBJECT:

14 DAY TWO LEVEL ACUTE ORAL RAT TOXICITY TEST USING EVS

SAMPLE # 11268622, BIOMED SAMPLE NUMBER 6652

METHODS, RESULTS AND CONCLUSIONS:

The sample was tested for its toxicity to male albino Sprague Dawley rats. Testing was in accordance with guidelines in the State of Washingtons Biological Testing Methods DOE 80-12, revised July, 1981. The rats were gavaged with the sample at a desage of 5g/kg and 0.5g/kg.

At the 5g/kg dosage all test animals were dead within one hour and fifteen minutes. There were no mortalities at the 0.5g/kg level during the test period. All animals were healthy and showed substantial weight gains at the time of termination. Due to its toxicity, BioMed Sample Number 6652 is considered to be a dangerous waste in rats.

If you have any further questions or comments, please do not hesitate to contact us.

Respectfully submitted,

Steven Lock

Fisheries Biologist

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INDUSTRY/TOXICANT EVS Consultants/a BIOMED RESEARCH LABORATORIES, INC. 1 6652 ADDRESS 3335 East Lake Ave Sectile, WA ANALYST STOVER LOCK BEGINNING TIME & DATE 12/29/86 2:00 pm COLLECTOR DATE SAMPLE COLLECTED 12/10/86 ENDING TIME & DATE ///2/87 TEST ORGANISM SOCIETY DAWLEY Albino 8 DOSAGE LEVEL 0,591 OBSERVATIONS AND DATES 14 KC 4HR 12/20 31 RAT # 1526 255 269 296 1.3 of oh oh \$ 1527 222 240 261 1.2 = 1528 247 247 262 1.2 1529 | 728 | 233 | 258 1.1 1530 266 763 281 1.3 237 244 263 1.2 1532 257 26/2861.3 1533 248 256 275 1.2 1534 253 244 256 1.3 1535 260 262 282 13 V

Client- Hart (1005) E.V.S. Project 8- 2/23/-03 Work Order 8- 870/63		E.V.S. Analyst(s)- RRILWI EC
identification- DS 3 1:1 (Henchress Amount Received- 21/4 Date Collected- April 20, 1987 ph- Dissolved Oxygen (mg/1)- Conductivity (umhos/cm)-	Helyested)	Bioassay Type- 96 hour War Hir TT Test Initiation Date- My L8, 1987
Other- Fresh Water (dechlorinated)- Salt Water (Burrard Inlet)- Dissolved Oxygen (mg/1)- Conductivity (umhos/cm)- Hardness (mg/1 as CaCO ₃)- That (mg/1 as CaCO ₃)- Salinity (mg/1 as CaCO ₃)- Salinity (mg/1 as CaCO ₃)- Other- LEP quide lineo TEST CONDITIONS Temperature (°C)- pH Range- Dissolved Oxygen Range- Conductivity Range- Aeration (7.5 cc/min./1)- Photoperiod (L:0-in hours)- Hardness (mg/1 as CaCO ₃)- 112- 74 - 9./ Photoperiod (L:0-in hours)- 14:10 No. Fish/Test Volume- Fish Loading Density (g/1)- O. 21	. hardnoss	TEST SPECIES Rainbow Trout- Threespine Stickleback- Daphnia (D. magna)- Amphipod (R. abronius)- Other- Of-100 mg/L (Ca(C3)
Broassay Results - 96 har UDCE	H.W T.T. E	hiszification =

Certified By- Locurea E.V.S. Consultants Ltd.

E.V.S. CONSULTANTS

DATE COLLECTED 1/4 (HAROLESS ADJECTED)											ACUTE LETHALITY BIOASSAY DATA												E.V.S. PROJECT NO. 27251-05											
DATE	COLLEC	TED	nla																						١	NOR	K O	RDER	NO	_87	<u>1016</u>	3 و		
DATE NO. (MIL)					 -			ENT 96		(VIVA	AL.		DIS		VED mg/L		rgen	TEMPERATURE (°C)							pΗ			COND (um	OUCTIV		AL mg	g/L	mg	ARO
L AB NO.	TIME	FISH/ VOL.	CONC.	1	2	4	8	18	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	96		0	96	0	96
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EAN FISH LENGTH (mm) 32 RANGE 27 . 35

0.32 RANGE 0.18 - 0.40

MEAN FISH WEIGHT

DATA VERIFIED BY & ROUSSELE.V.S. CONSULTANTS

TATE May 20, 1987

E.Y.S. CONSULTANTS ACUTE LETHALITY BIOASSAY RECORD

)	Client- Hart- Crowser	E.V.S. Analyse(s) - WINLIES
	E.V.S. Project 1- 21231-03	
	Work Order 1- 870268	
	SAMPLE	
	Identification- DS-3 (100 ppm)	Bloassay Type- WDOG HUTT Duggm
	Amount Received-	Test Initiation Date- 1: 23/87
	Date Collected- n/q	1000 11100 1010 1
	Date Received- requested in 16/87	
	рн-	
	Dissolved Oxygen (mg/l)-	
	Conductivity (umhos/cm)-	
6.下達	Other-	
~ !"		
lmar: s	DILUTION AND CONTROL MEDIUM	TEST SPECIES
	Fresh Water (dechlorinated)-	Rainbow Trout-
C3	Salt Water (Burrard Inlet)-	Threespine Stickleback-
n	pH <u> </u>	Daphnia (<u>D. magna</u>)-
***	Dissolved Oxygen (mg/1)- 10.5	Amphipod (R. abronius)-
1	Conductivity (umhos/cm)-	Other-
-	.Hardness (mg/l as CaCO ₃)- 118	
	Alkalinity (mg/l as CaCO ₃)- 85	
Market.	Salinity (0/00)-	> 11 1
\bigcirc	Other- * Responder adjusted to 3	130 mg/4 (as (a(0,)) Hardross
-	and 278 mg/ (as la CO3)	130 mc/L (as (b(0,) Hadross Alkalikit, Az per EPA guidelines thesh water.
	TEST CONDITIONS TO PECONOTIFIED	vest wour.
	Temperature (°C)- 12±/	
	pH Range- <u>7.7 - 8.1</u>	
	Dissolved Oxygen Range- 7.6-10.5	
	Conductivity Range-	· ·
	Aeration (7.5 cc/min./1)-	
	Photoperiod (L:D-in hours)- (4:10	
	No. Fish/Test Volume- 10/201	
	Fish Loading Density (g/1)- 0.39	
	Other-	
	Bioassay Results - 2/30 Lish mor	talities @ 100 nom
		hudion = none
		2 3E-15 E.Y.S. Consultants Ltd.

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E.V.5.	CONSULTANTS
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N FISH LENGTH (mm) 39 RANGE 36-43 N FISH WEIGHT (g) 077 RANGE 069-049

TE July 10, 1987.



EVS Consultants 2335 Eastlake Ave. Seattle, WA 98102

Attn: Bob Dexter

SUBJECT:

1.1

14 DAY ACUTE ORAL RAT TOXICITY TEST USING EVS sample

BIOMED SAMPLE NUMBER 7732. DS-3 1:1 DSSF, project #21331-03.

METHODS, RESULTS AND CONCLUSIONS:

The sample was tested for its toxicity to male albino Sprague Dawley rats.

Testing was in accordance with guidelines in the State of Washingtons.

Biological Testing Methods DOE 80-12, revised July, 1981. The rats were gavaged with the sample at a 5 gm./kg. dosage level.

There was one mortality during the test period. All other animals were healthy and showed substantial weight gains at the time of termination. Due to its lack of toxicity, BloMed Sample Number 7732 is not considered to be a dangerous westering the sample Number 7732.

If you have any further questions or comments, please do not hesitate to contact us.

Respectfully submitted

Steven Lock

Fisheries Biologist

PATA SELLI FOR ACUI. RAL RAT TOATCITI TEST

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E.V.S. CONSULTANTS ACUTE LETHALITY BIOASSAY RECORD

Client- Heat - Cresses E.V.S. Project #- 21231-03 Work Order #- 870163	E.V.S. Analyse(s)- RRILWIEW
SAMPLE Identification- S-14 1:1 (HAIZDAE) Amount Received- 2 1kg Date Collected	Test Initiation Date- 14,13/82
(Other-	
Fresh Water (dechlorinated)- Salt Water (Burrard Inlet)-	TEST SPECIES Rainbow Trout- Threespine Stickleback-
TA 7.9	Daphnia (<u>D. magna</u>)-
TDissolved Oxygen (mg/1)- 10.1	Amphipod (R. abronius)-
Conductivity (umhos/cm)-	Other-
Hardness (mg/1 as CaCO ₃)-	
Alkalinity (mg/1 as CaCO ₃)- 60 Salinity (°/00)- nla Other- *unter recombinated to hardn on ap per EPA gurdelines	ess = 100 mg/n (Ca(Oz)
TEST CONDITIONS	
Temperature (°C)- 12±1.0	
pH Range- 7.5-8 [
Dissolved Oxygen Range- 93-10-1	
Conductivity Range-	
Aeration (7 5 cc/min /1)	
Photoperiod (L.D-in hours)-	
No. Fish/Test Volume- 10/252.	
Fish Loading Density (g/1) - U.21	
Other-	
Bloassay Resules. 96 hour WDCE	HWTT classification =

APP 3E-19

E.V L Consultants Ltd.

Vel 16 mentalities/30 fish a 102-11/L

APP 3E-20

SAMPLE _	D5-19	1:1	Hundress	<u>adjusted)</u>
DATE COL	LECTED _			

ACUTE LETHALITY BIOASSAY DATA

E.V.S. PROJECT NO. 2/231-03

WORK ORDER NO. _ 870163

	TEST DATE	NO.	(('~'						SUR		L		DIS		VED mg/L		'GEN		TEM	PER/ (°C)	ATU	RE.			рΗ			COND (vm	UCTI hos/c	AL mg	K ML	H	
LAB NO.	TIME	FISH/ VOL.	CONC.	-	2	4	8	18	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	24	48	72	96	0	96	0	96	0	9
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COMMENTS * all Noman fish a stressed; discland; discreted; lass of equilibrium 8

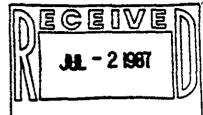
MEAN FISH LENGTH (mm) 32 RANGE 27 - 35

EAN FISH WEIGHT (9) 0.32 RANGE 0.18 - 0.40

DATA VERIFIED BY & KOUNSON E.V.S. CONSULTANTS

DATE May 20, 1987





EVS Consultants 2335 Eastlake Ave. Seattle, WA 98102

Attn: Bob Dexter

SUBJECT:

1.0

14 DAY ACUTE ORAL RAT TOXICITY TEST USING EVS sample

BIOMED SAMPLE NUMBER 7733. DS-19 1:1 DSSF, project #21231-03.

- METHODS, RESULTS AND CONCLUSIONS:

The sample was tested for its toxicity to male albino Sprague Dawley rats.
Testing was in accordance with guidelines in the State of Washingtons
Biological Testing Methods DOE 80-12, revised July, 1981. The rats were
gavaged with the sample at a 5 gm./kg.dosage level.

There were five mortalities during the test period. All other animals were healthy and showed substantial weight gains at the time of termination. Due to its toxicity, BioMed Sample Number 7733 is considered to be a dangerous waste in rate.

If you have any further questions or comments, please do not hesitate to contact us.

Respectfully submitted.

Steven Lock

Fisheries Biologist

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TEST O	RGANI	sm <u>/</u>	Sal	વવા	e L	awle	y A	biào	DOSA	GE LE	VEL_	59	lkg		/					
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RAT #	0	7	14	1 1	4HR	6/18	119	70	71	22	23	241	25	76	27	28	29	2	3/1	OMMENT
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APPENDIX 3F

PILOT-SCALE TEST REPORT--EVALUATION OF GROUT PROCESSING PARAMETERS

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APPENDIX 3F

PILOT-SCALE TEST REPORT EVALUATION OF GROUT PROCESSING PARAMETERS

This appendix provides information concerning pilot-scale testing of grouting equipment. It was conducted using a nondangerous simulated waste form. The test was intended only to verify the general principles related to mixing cementitious materials with liquid wastes.

The test results indicate that laboratory tests correlate with large-scale field results.

This appendix (Pacific Northwest Laboratory Document No. PNL-6148) contains 91 pages.

DOE/RL 88-27 Rev. 1, 01/17/90

Hanford Grout Technology Program

Pilot-Scale Grout Production Test with a Simulated Low-Level Waste

C. L. Fow D. H. Mitchell R. L. Treat C. R. Hymas

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May 1987

Prepared for the U.S. Department of Energy under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute



PILOT-SCALE GROUT PRODUCTION TEST WITH A SIMULATED LOW-LEVEL WASTE

C. L. Fow D. H. Mitchell R. L. Treat C. R. Hymas

May 1987

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Prepared for the U.S. Department of Energy under Contract DE-ACO6-76RLO 1830

Pacific Northwest Laboratory Richland, Washington 99352

SUMMARY

Plans are underway at the Hanford Site near Richland, Washington, to convert the low-level fraction of radioactive liquid wastes to a grout form for permanent disposal. Grout is a mixture of liquid waste and grout formers, including portland cement, fly ash, and clays. In the plan, the grout slurry is pumped to subsurface concrete vaults on the Hanford Site, where the grout will solidify into large monoliths, thereby immobilizing the waste. A similar disposal concept is being planned at the Savannah River Laboratory site. The underground disposal of grout was conducted at Oak Ridge National Laboratory between 1966 and 1984 (Dole 1985).

Design and construction of grout processing and disposal facilities are cunderway. The Transportable Grout Facility (TGF), operated by Rockwell Hanford Operations (Rockwell) for the Department of Energy (DOE), is scheduled to grout Phosphate/Sulfate N Reactor Operations Waste (PSW) in FY 1988. Phosphate/Sulfate Waste is a blend of two low-level waste streams generated at Hanford's N Reactor. (The N Reactor produces special nuclear materials, and its byproduct steam is used to generate electricity.) Other wastes are scheduled to be grouted in subsequent years.

Pacific Northwest Laboratory (PNL) is verifying that Hanford grouts can be safely and efficiently processed. To meet this objective, pilot-scale grout process equipment was installed. The pilot-scale process equipment can produce grout at a rate of up to 25% of the maximum rate planned for the TGF.

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On July 29 and 30, 1986, PNL conducted a pilot-scale grout production test for Rockwell. During the test, 16,000 gallons of simulated nonradioactive PSW were mixed with grout formers to produce 22,000 gallons of PSW grout. The grout was pumped at a nominal rate of 15 gpm (~25% of the nominal production rate planned for the TGF) to a lined and covered trench with a capacity of 30,000 gallons. Emplacement of grout in the trench will permit subsequent evaluation of homogeneity of grout in a large monolith. The production of a

22,000-gal monolith in a trench also permitted determination of curing characteristics, reabsorption of separated liquid, degree of cracking, and temperature rise expected with monolithic disposal.

The principal process components—the grout mixer and the grout pump—are very similar to those planned for the Transportable Grout Facility Equipment (TGE). The pilot—scale test permitted evaluation of the performance of the mixer and pump, their flush requirements, and their reliability. In addition, representatives of the engineering firm commissioned to design and construct the processing equipment modules of the TGF observed the test to gain experience with processing grout.

The test was very successful; major conclusions follow:

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C. C

- The continuous grout mixer and grout pump performed reliably, producing grout with acceptable properties.
- The adiabatic grout temperature rise was at least 37°C, and probably higher.
- The flow angle of grout in the trench averaged 1.5°. A similar flow angle can be expected in the disposal vaults with grouts of the same rheological properties.
- The degree of cracking of grout in the trench was minimal, reducing concern over the effect of additional surface area on the performance assessment of this disposal method.
- The separated liquid that collected on the surface of the grout monolith was totally reabsorbed in 30 days. If the TGF operates under similar conditions (grout rheology and ratio of flush water to grout volume), total reabsorption can be expected.

Analyses of samples of grout, separated liquid, dry blend, and simulated PSW taken during and after the pilot-scale tests were in progress at the time this report was prepared. A future report will discuss the homogeneity of grout in the monolith and the properties of the samples collected.

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1.0 INTRODUCTION

Plans are underway at the Hanford Site near Richland, Washington, to convert the low-level fraction of radioactive liquid wastes to a grout form for permanent disposal. Grout is a mixture of liquid waste and grout formers, including portland cement, fly ash, and clays. In the plan, the mixture is pumped to subsurface concrete vaults on the Hanford Site, where the grout will harden into large monoliths, thereby immobilizing the waste. A similar disposal concept is being planned at the Savannah River Laboratory site. The underground disposal of grout was conducted at Oak Ridge National Laboratory (ORNL) between 1966 and 1984.

Design and construction of grout processing and disposal facilities are underway. The Transportable Grout Facility (TGF), operated by Rockwell Hanford Operations (Rockwell) for the Department of Energy (DOE), is scheduled to grout Phosphate/Sulfate N Reactor Operations Waste (PSW) in FY 1988. Phosphate/—Sulfate Waste is a blend of two low-level waste streams generated at Hanford's CN Reactor. (The N Reactor produces special nuclear materials, and its abyproduct steam is used to generate electricity). Other wastes are scheduled to be grouted in subsequent years.

The Transportable Grout Facility includes the Dry Materials Receiving and -Handling Facility (DMRHF) and the Transportable Grout Equipment (TGE). Cement, -clays, and fly ash will be received, stored, and blended at the DMRHF. The chlended material will be loaded and shipped to the TGE. In the TGE, the dry material will be mixed with the liquid waste to form grout. The TGE consists of seven transportable modules: 1) dry blend module, 2) mixer, pump and liquid collection module, 3) control room module, 4) electrical equipment module, 5) heating, ventilating and cooling module, 6) standby generator module, and 7) additives and decontamination module.

The grout produced in the TGE will be pumped to subsurface disposal vaults. The vaults are concrete enclosures with a 1.4 million gallon capacity. Vault dimensions are 125 feet long by 50 feet wide by 35 feet deep. Vaults will contain a liner system consisting of a drainage net between two layers of 60-mil-thick, high-density polyethylene.

Pacific Northwest Laboratory (PNL) is verifying that Hanford grouts can be safely and efficiently processed. To meet this objective, pilot-scale grout process equipment was installed. The pilot-scale process equipment can produce grout at a rate of up to 25% of the maximum rate of 70 gpm planned for the TGF. Since 1984, PNL has performed seven major tests with pilot-scale equipment, producing simulated PSW grout to evaluate the performance of process equipment and grout behavior.

This report presents the results of a 24-hr test of the pilot-scale grout process conducted on July 29 and 30, 1986. Results of earlier, unreported pilot-scale tests are also cited for comparison.

1.1 OBJECTIVES OF THE PILOT-SCALE TEST

The three objectives of the pilot-scale test were: 1) to determine the homogeneity of the grout produced under conditions similar to those planned for the TGF, 2) to evaluate performance of candidate grout processing equipment for the TGF, and 3) to evaluate properties of grout that was produced during continuous operation over an extended time period and disposed in a large trench.

Because of the extended duration of the test, process data were obtained that will be useful in the design of the full-scale grout process equipment and in the development of the operating procedures for the TGF. Additionally, observations of grout behavior and measurements of grout properties in a disposal system similar to the proposed vaults will support the design of the disposal vaults.

1.2 SCOPE OF PILOT-SCALE TEST

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A large grout monolith (approximately 22,000 gallons) was produced using simulated nonradioactive PSW and a cementitious blend of dry materials based on a formulation developed by Oak Ridge National Laboratory. $^{(a)}$ The test utilized

⁽a) Letter Report McDaniel, E. W. et al. Grout Formulation Studies with Hanford Facility Waste: An Executive Summary. Oak Ridge National Laboratory, Oak Ridge, Tennessee (September 1984).

pilot-scale grout processing equipment similar to those proposed for the TGE. Grout was produced at a rate of 15 gpm.

The simulated PSW contained nonradioactive trace components simulating corrosion products. The dry blend of grout formers used in the test was produced at the Dry Materials Receiving and Handling Facility (DMRHF). The DMRHF is an integral part of the TGF.

During grout production, data were taken to evaluate equipment performance, homogeneity of the grout produced under conditions similar to those planned for the Transportable Grout Facility Equipment (TGE), equipment flush requirements, and grout physical and rheological properties. Grout physical and rheological properties were evaluated at both the mixer discharge and the trench discharge to examine the effect of shear in the pipe on these properties.

The grout was disposed in a 30,000-gal capacity trench that had features similar to the vaults to be used for disposal of actual PSW grout. The trench was lined with 60-mil-thick high-density polyethylene (HDPE). A plastic cover was placed over the trench to prevent entry of foreign material and evaporation of water from the grout. Access ports in the cover allowed operators to insert sample tubes, observe grout behavior in the trench, and withdraw samples.

During the first month after the grout was produced, samples of separated liquid on the grout surface were collected three times per week and analyzed for pH, heavy metals, and organic carbon. Grout temperatures were monitored at least every day. The volume of separated liquid was monitored until it was completely reabsorbed 30 days after the grout was produced. Grout core samples were extracted from the monolith on the 28th day and stored in vapor-tight containers for subsequent physical and chemical tests.

In the fall of 1986, the grout monolith was insulated and covered with an additional layer of plastic. Temperature measurements in the monolith will continue as long as the effort is justified. After approximately 8 months, the monolith will be examined to determine the frequency of crack development in the grout. The monolith will then be dug up and permanently disposed in a landfill.

A sampling plan for the pilot-scale test was prepared to provide a statistical basis for determination of the homogeneity of the grout and other grout properties. The plan called for tests on the simulated waste, the dry blend, the fresh and cured grout, and the separated liquid. These analyses will be used to assess the uniformity of the grout in the monolith and to provide data on the physical properties needed to assess the long-term performance of disposed grout.

This report discusses the operational aspects of the pilot-scale test in detail. Whenever possible, discussions are included that relate the performance observed during the pilot-scale test to the performance expected in the TGF.

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œ چ The pilot-scale equipment and trench are described in Chapter 2.0. The procedures for preparation of the dry blend, preparation of simulated PSW, and sampling are discussed in Chapter 3.0. Chapter 4.0 reports results of the pilot-scale test. This chapter includes evaluations of three areas: 1) equipment performance, 2) flush system performance and requirements, and 3) behavior of grout in the trench. Chapter 4.0 also presents the results of laboratory rheology tests performed prior to and during the test. Chapter 5.0 summarizes conclusions from the pilot-scale test, as well as recommendations for the TGF design and for improvements in the pilot-scale system.

2.0 DESCRIPTION OF EQUIPMENT AND TRENCH

This chapter describes the pilot-scale grout processing equipment and disposal trench. The equipment is sized to process grout at up to 25% of the 70 gpm maximum production rate planned for the TGF. A schematic of the system is shown in Figure 2.1, each component is discussed.

2.1 WASTE SUPPLY

Simulated waste was pumped to the grout processing equipment from a 23,000-gal carbon steel tank. (Section 3.1 describes the simulated waste preparation and composition.) Two parallel centrifugal pumps (one for backup) supplied the waste to the grout mixer. Part of the waste stream was recirculated back to the tank. The recirculation loop was designed to prevent complete stoppage of flow in the event the waste flow to the mixer was stopped. A second recirculation system served to suspend precipitated material in the complete. This system consisted of a 1-horsepower pump and a flow distribution header with 170 nozzles. The distribution header was located on the floor of the tank.

The waste flow rate was controlled with a manually operated gate valve.

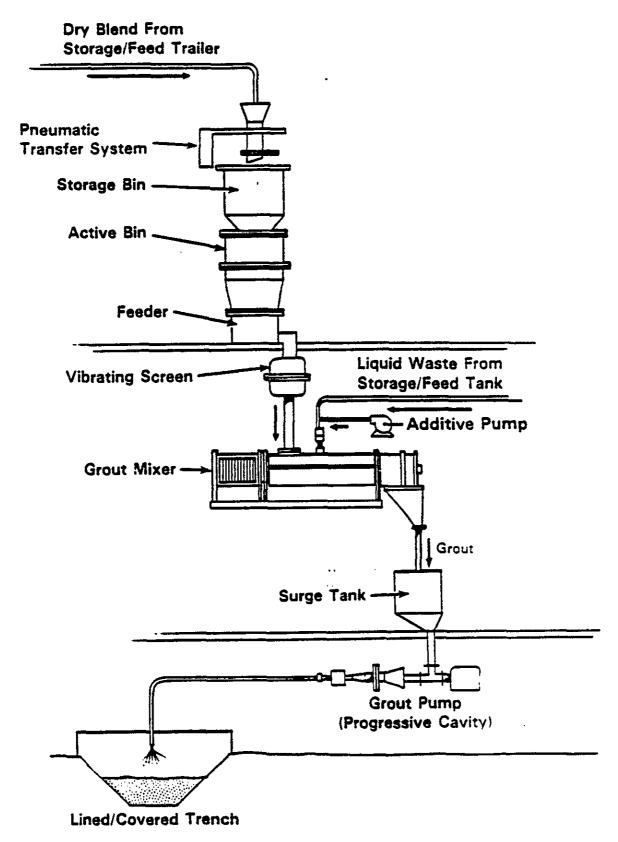
Flow rate instrumentation is described in Section 2.7.2. The temperature of the waste was measured near the inlet to the grout mixer and recorded on a datalogger.

The synthetic waste was prepared in batches in an agitated 4,200-gal stainless-steel tank and then pumped to the feed tank. Section 3.1 discusses the procedures used to prepare the waste.

Tributyl phosphate (TBP), a deaerating agent, was added to the waste at the mixer inlet. A teflor diaphragm pump was used to meter the TBP at a rate equal to 0.02% of the waste volumetric rate.

2.2 DRY BLEND FEED

Components of the dry-blend feed system include the supply trailer, the trailer-to-storage bin transfer system, the storage bin/baghouse, the active bin/feeder, and the scalping screen.



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FIGURE 2.1. Schematic of Pilot-Scale Process

Dry blend (a mixture of 41 wt% portland cement I & II, 40 wt% Class F fly ash, 11 wt% Attapulgite-150 drilling clay, and 8 wt% Indian Red Pottery Clay) was supplied in trailers, each with a 1000-ft³ capacity (Figure 2.2). The dry blend was produced at the DMRHF. Three trailer loads were used during the test.

Using a vacuum system, dry blend was transferred in batches from the trailer unloading port to a storage bin with a 27-ft³ capacity. The storage bin contains a baghouse to separate the dry blend from the transfer air. (The transfer system and storage bin were manufactured by Vac-U-Max®.)

The storage bin is located above the active bin/feeder. The contents of the storage bin were automatically dumped to the active bin on a signal from

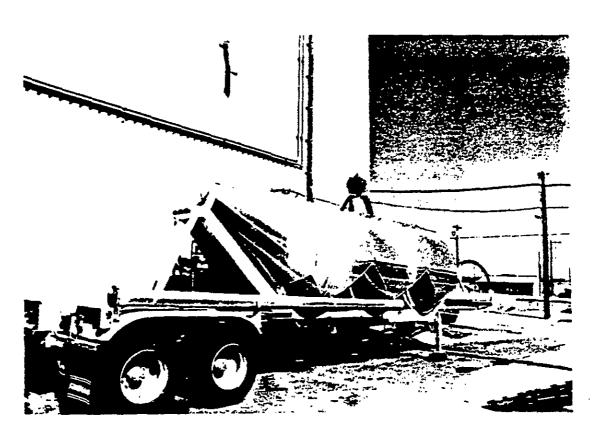


FIGURE 2.2. Dry Blend Supply Trailer

Tradename of Vac-U-Max, Belleville, New Jersey.

the feeder controller. Figure 2.3 shows the baghouse/storage bin, active bin/feeder, and vacuum transfer pump. The active bin has a capacity of 36.6 ft^3 and an active volume of 30 ft^3 . The feeder is an Acrison® gravimetric (augertype) feeder with a weight rate accuracy of 0.5% of the set point. When the feed bin weight reaches a predetermined low level, the feeder is automatically

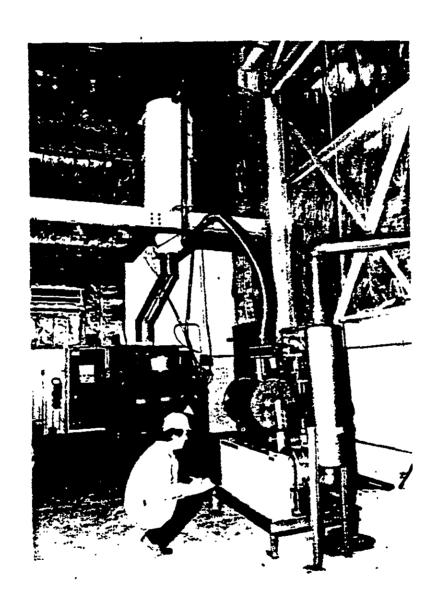


FIGURE 2.3. The Dry Blend Transfer/Feed System. Storage bin/baghouse is above the feeder; the transfer blower is in the foreground.

Tradename of Acrison, Inc., Moonachie, New Jersey.

switched to a volumetric mode and a valve between the feeder and the storage bin is opened for reloading. At this time, a vibrator and air pads on the storage bin are activated to promote the discharge of dry blend from the storage bin. The reload valve is closed when the weight reaches 90% of the feeder capacity.

Excellent feeder performance was demonstrated in pre-tests in support of the pilot-scale test. The pilot-scale feed system differs significantly from the gravimetric belt feed system planned for the TGF.

The feeder discharged into a Sweco vibrating screen. This 18-in. diameter scalping screen was designed to remove material greater than 0.20 inch from the dry feed. The oversize material is collected in a 5-gallon receiving drum. The TGE will also use a vibrating screen upstream of the continuous grout mixer.

2.3 MIXER

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Dry blend and simulated waste are combined in the mixer to produce a grout slurry. The grout mixer is a Teledyne Readco® 5.25-in. Continuous Processor (Figure 2.4). Dry blend and simulated waste enter at the top of the mixer. The grout discharges at the opposite end of the mixer to the pump surge tank. The mixer has a water spray system for flushing the dry-blend inlet and grout discharge sections. Figure 2.5 shows the interior of the mixer. Dry blend is introduced at the left end into the screw section. Liquid is introduced where the mixer paddles begin. The paddles are 1 inch wide and provide low-shear mixing of grout during mixer operation.

Mixing speed is adjustable from 50 to 270 rpm. For the pilot-scale test, the mixer was operated at 250 rpm, a speed that had been chosen based on results of tests that showed that slightly less dusting occurred at higher rpm's without measurable effects on the grout properties. The mixer has an adjustable discharge gate that can be used to adjust residence time of grout in the mixer. Partial closure of the discharge gate was also found to reduce dusting, probably due to the increase in residence time of the grout.

Tradename of Sweco, Inc., Los Angeles, California.

Tradename of Teledyne Readco, York, Pennsylvania.

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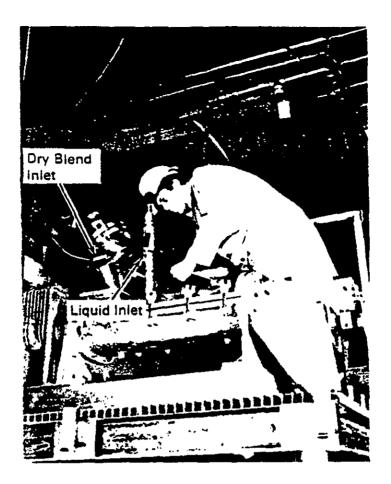


FIGURE 2.4. The Continuous Grout Mixer

The mixer that will be used for the TGE mixer will also be manufactured by Teledyne-Readco. The mixing paddle will be 7 inches long instead of 5.25 inches, and the mixer will have no adjustable discharge gate. The performance of the TGE mixer is expected to be very similar to that of the pilot-scale mixer.

The pilot-scale mixer discharges grout into a surge tank where operators can sample grout and measure the grout production rate. The surge tank is 19 inches in diameter and 20 inches deep, with a cone-shaped base. Screens in the surge tank are used to collect foreign material to prevent damage of the grout pump. The first screen is cylindrical (6 inches in diameter) with 0.1-in. openings. This screen catches any oversize material from the mixer. The second screen (with ~ 0.5 -in. openings) is at the base of the surge tank and



FIGURE 2.5. Internal View of Grout Mixer

provides protection in case items are accidentally dropped into the surge tank. These screens will not be used in the TGE because the surge tank will be a closed vessel. Also, the TGE pump is capable of passing much larger particles without damaging the pump. Because the pilot-scale surge tank is open, however, there is greater potential for foreign material to reach the pump.

2.4 GROUT PUMP

The grout pump is a two-stage progressive-cavity pump (Figure 2.6) with an ethylene-propylene-diene-monomer (EPDM) stator. The pump seal is a water-lubricated packing gland. The pump-speed is manually controlled with a digital-setpoint speed control and a digital rpm indicator to maintain a constant level of grout in the surge tank. The pump amperage and speed are recorded on the datalogger. A progressive-cavity grout pump will also be used as the TGE pump; however, it will have more stages to produce the higher pressure required to pump grout over longer distances to vaults.

2.5 PIPING

The grout pump discharges grout into a 1-in. Schedule 40, carbon steel pipe that runs to the trench. The piping was sized to maintain turbulent flow at the planned production rate of 15 gpm assuming typical rheological

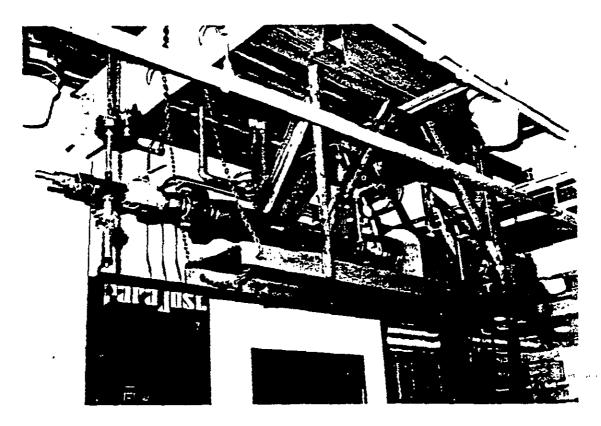


FIGURE 2.6 Progressive-Cavity Grout Pump

properties of PSW grout. In the pilot-scale test, the piping had an equivalent length of 155 feet and contained eight long-radius (4-in.) elbows. The long radius elbows were used to minimize both erosion rates and the likelihood of developing "dead spots" where grout solids could collect. Grout was discharged vertically to the trench at a single point through the trench roof.

2.6 TRENCH

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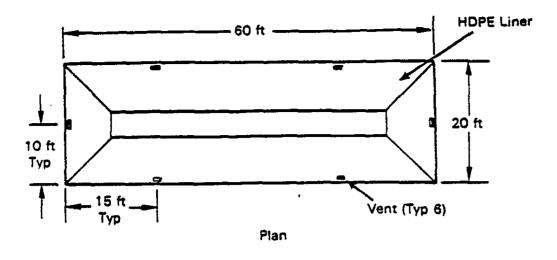
The pilot-scale trench was 8 feet deep with 45° side slopes. The dimensions at the top of the trench were 60 feet by 20 feet (Figure 2.7). The capacity, excluding 1 foot of freeboard, was 30,000 gallons. The trench was lined with a sheet of 60-mil-thick HDPE. The trench design for the pilot-scale test was based on the then-current design for disposing of the radioactive grouted waste. Since that time, the disposal concept evolved from trenches to disposal vaults. The current vault design includes vertical side walls, top dimensions of 125 feet by 50 feet, and a liner system consisting of a drainage net sandwiched between two layers of 60-mil-thick HDPE.

The HDPE for the pilot-scale trench was procured from and installed by Northwest Linings; (a) the HDPE liner was manufactured by National Seal. (b) The liner was seamed, where practical, using a double-wedge, heat-welded seam method. An extrusion weld process was used where the heat-welder could not be used. Figures 2.8a and b show schematics of the types of seams made by these two processes. Figure 2.9 shows the trench during liner installation.

Double-weld seams were tested for leaks by sealing each end of a seam and then inserting a needle in the air space between the two welds. The seams were pressurized to about 30 psi with air, and the bleed rate was observed. The criterion for unacceptable seams was a bleed rate greater than 4 psi over 15 minutes; however, all seams tested by this method were acceptable. Some of the extruded seams and patches were tested by a vacuum box inspection method; however, due to the uneven subgrade, this technique was not very effective and therefore was not used extensively. Instead, visual examinations and "pick"

⁽a) Northwest Linings, Bellevue, Washington.

⁽b) National Seal, Palantine, Illinois.



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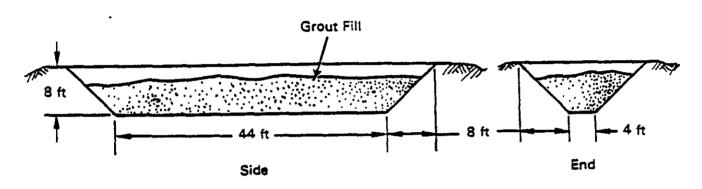
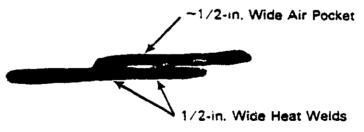


FIGURE 2.7. Schematic of Grout Trench

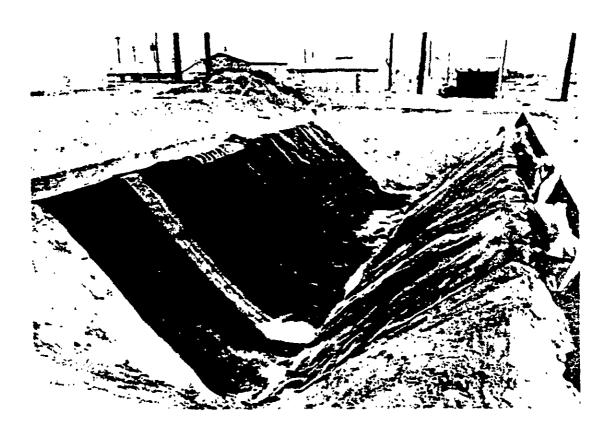


a) Double Wedge Heat-Welded Seam



b) Extrusion Fillet Weld Seam

FIGURE 2.8. Cross-Section of HDPE Seams Used in the Trench Liner



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FIGURE 2.9. Trench Under Construction

tests were performed. (In a pick test, a pointed object, e.g., a nail, its run along the edge of the seam to determine whether the bond is continuous between the two layers.)

A wood-frame roof was constructed over the trench. A 20-mil-thick polyvinyl chloride (PVC) vapor barrier was placed over the wood frame. Several access ports were built on the top and sides of the cover to permit sampling of grout, observation of grout flow angle, and estimation of separated liquid volume. Marks on the trench liner were used to measure grout flow angles and to estimate separated liquid volume. Figure 2.9 shows the trench under construction and Figure 2.10 shows the completed trench cover with samplers (vertical pipes) installed after the test.

The grout discharge nozzle was located near one end of the trench so that grout flowed approximately 50 feet under conditions similar to those expected

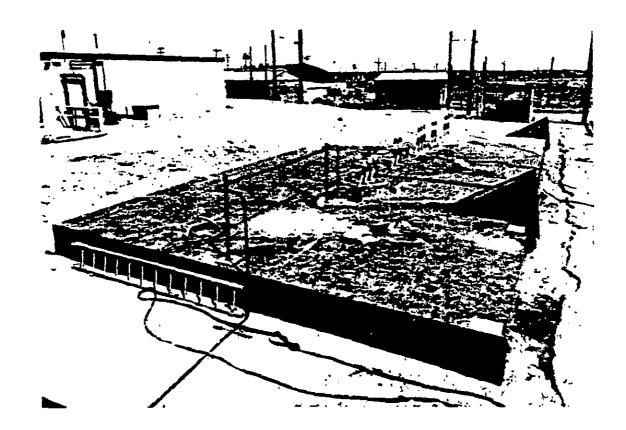


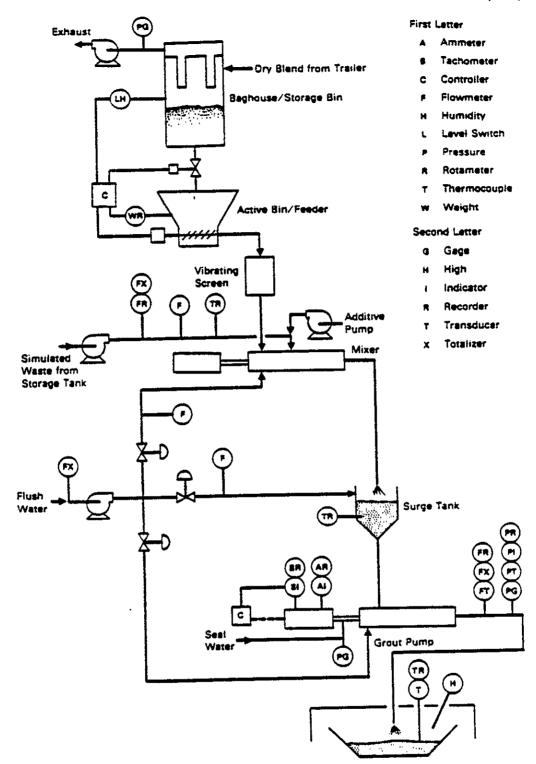
FIGURE 2.10. Completed Trench Cover

in vaults for PSW grout. (Grout would flow a maximum distance of 67 feet in the current design for a disposal vault.)

During the week prior to grout production, the trench liner was washed to remove dust and debris that had collected during construction of the cover. The soil in the anchor trenches for the liner was soaked with water to minimize its capacity to absorb moisture from the trench vapor space.

2.7 PROCESS INSTRUMENTATION

Process instrumentation is depicted in Figure 2.11. The datalogger recorded outputs from most instruments in 15-min. intervals. The various instruments used in the test are described below.



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FIGURE 2.11. Process Instrumentation

2.7.1 Temperature Measurement

Temperatures of waste, grout, and ambient air were measured at several locations during the test: 1) waste at the inlet to the mixer, 2) grout in the surge tank, 3) grout at 35 locations in the grout trench, and 4) ambient air temperature outside the trench. Measurement of the trench temperatures continued after the end of grout production and is ongoing as of the publication date of this report. All temperatures were measured using Type K thermocouples with stainless steel sheaths.

The schematic of the trench showing thermocouple locations is provided in Figure 2.12. Each thermocouple was numbered; a total of 35 thermocouples were placed. Three thermocouple arrays were located vertically in the center of the trench 2, 17, and 40 feet from the discharge pipe. A fourth array was positioned along the side slope 17 feet from the nozzle, and the fifth was located between the vertical array and the array on the slope. This arrangement of the thermocouples was designed to permit analysis of heat distribution throughout the trench. The thermocouples were strapped to a 0.5-in. tube with the ends of the thermocouples spaced at 1-ft intervals from the trench floor. The lowest thermocouple of each array was positioned 2 inches above the floor of the trench. The tips of the thermocouples were bent 2 inches away from the tube to reduce the heat sink effect on the measurement. The tubes that supported the thermocouples were anchored to steel plates on the floor of the trench to prevent displacement of the thermocouples by the flowing grout.

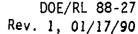
2.7.2 Flow Measurements

The waste flow rate was measured with a magnetic flowmeter and a rotameter. Grout flow rate in the discharge piping was measured with a magnetic flowmeter. Outputs from the magnetic flowmeters were recorded on the datalogger.

2.7.3 Pressure Measurement

Grout pressure at the pump discharge was measured using an Iso-Spool® system, which transmits pressure across a rubber diaphragm to both a liquid-filled

Tradename of Ronningen-Petter Div., Dover Corp. Portage, Michigan.



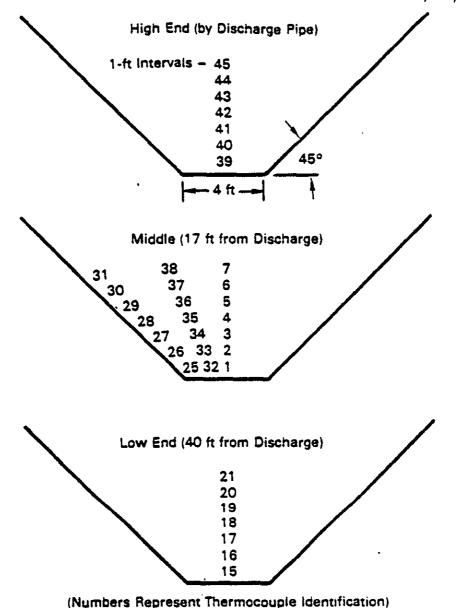


FIGURE 2.12. Schematic of Trench Thermocouples

pressure gauge and a transducer. The datalogger recorded the transducer output and was programmed to shut the pump down if the pressure exceeded 125 psig.

2.7.4 Miscellaneous Measurements

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Pump amperage and rpm were recorded on the datalogger. Data from the gravimetric dry blend feeder (weight loss rate) were recorded on the datalogger. Relative humidity in the trench was measured using a hand-held meter.

3.0 MATERIAL PREPARATION AND SAMPLING PROCEDURES

This chapter describes the procedures used to prepare the simulated waste and dry blend. It also describes the sampling procedures used during the test.

3.1 SYNTHETIC WASTE PREPARATION

The simulated waste consists of equal volumes of aqueous phosphate and sulfate wastes. Phosphate waste is generated during the decontamination of N Reactor. This waste's principal ingredient is phosphoric acid, which is neutralized with sodium hydroxide prior to storage. Sulfate waste results from the regeneration of ion-exchange columns used to clean water in spent fuel storage basins at N Reactor. The major component of sulfate waste is sulfuric acid that has been neutralized with sodium hydroxide. Included in the sulfate waste is a third minor stream called "sandfilter backwash" which is mixed with the sulfate waste at a ratio of 50 kg of sludge per million liters of sulfate waste. The sandfilters remove solids from the process stream prior to the ion exchange columns.

Batches of simulated phosphate and sulfate wastes were prepared in an agitated stainless steel tank using the formula in Table 3.1 and then pumped to the waste feed tank. Prior to grout production, the synthetic waste was analyzed for pH and major cations and anions. Table 3.2 compares the target concentrations of major species to the measured concentration values. As shown, there is reasonable agreement between the target and the measured compositions. The measured calcium concentration was greater than the target value due to calcium in the tap water used to make up the waste. The iron level was lower than the target value, perhaps due to sampling deficiency (i.e., the sample was deficient in precipitate containing iron). The chloride level was higher than the target value, presumably due to contamination in the sodium hydroxide and/or sodium sulfate. However, in these concentration ranges, the discrepancy in the actual values as compared to the target values is not expected to affect leach resistance or curing of the grout.

Trace chemicals such as Cr, As, Se, etc. were added in amounts that corresponded to analyses conducted on actual waste samples. The diethylthiourea

TABLE 3.1. Formulas for Preparation of Phosphate and Sulfate Wastes

	Component	Phosphate Waste (4000-Gal Batch)	Sulfate Waste (4000-Gal Batch)
	Tap water Turco 4512A-17	25,000 lb 102 gal	25,000 1b 0
	(without inhibitor) 1,3-Diethyl 2-thiourea Na ₂ SO ₄	1,817 g 0	0 68,310 g
	As ₂ 0 ₃	. 0.061 g	0.08 g
	BaCO3	1.12 g	8.18 g
	Cd(NO ₃)2.4H20	0.1 g	2.271 g
	Cr(NO3)3.9H20	167 g	1,105 g
	Hg(NO ₃) ₂	0.14 g	0.40 g
	H ₂ SeO ₃	0.074 g	0.098 g
()	AgNO ₃	0.288 g	1.97 g
O	CuSO4+5H20	4.088 g	18.2 g
Mene a	Fe(NO ₃) ₃ •9H ₂ O	17,411 g	1,696 g
(Fe ₂ (SO ₄) ₃	1,968 g	24,224 g
ru 	MnS0 ₄ •H ₂ 0	999 g	115 g
	ZnS0 ₄ •7H ₂ 0	47 g	2,574 g
(7)	Pb(NO ₃) ₂	1.075 g	19.7 g
3000	Ni (NO3)2+6H2O	139 g	227 g
National Property lies	Ca(NO ₃) ₂ •4H ₂ O	197 g	0
*****	CaSO4.0.5H20	0	1,105 g
C	KNO3	0	303 g
()°	A1 (NO ₃) 3.9H20	0	3,634 g
	NaF	394 g	1,332 g
	NaC1	545 g	999 g
	NaOH flakes	535.2 kg	22.2 kg
	Tap water	to 4000-gal level	to 4000-gal level

TABLE 3.2. Target and Measured Concentrations of Simulated PSW

Component	Target Concentration, ppb	Measured Concentration, ppb (one analysis)	
Cations			
A1	8,600	8,100	
As	4	below detection limit of 80	
Ag	. 50	not analyzed	
Ва	200	below detection limit of 2	
Ca	11,000	22,000	
Cd	30	below detection limit of 4	
Cr	5,500	3,500	
Cu	200	500	
Fe	329,000	170,000	
Нд	10	not analyzed	
K	3,900	below detection limit of 300	
Mn	12,000	8,400	
Na	no target	12,600,000	
Ni ·	2,400	1,500	
РЬ	400	below detection limit of 60	
Se	3	not analyzed	
Si	no target	8,900	
Zn	19,800	17,000	
<u>Anions</u>		•	
C 1	31,000	220,000	
F	26,000	<50,000	
P0 ₄	13,700,000	11,600,000	
NO ₃	385,000	400,000	
504	2,200,000	2,000,000	
рН	11.5 - 12.5	12.41	

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is a corrosion inhibitor that is added to the decontamination agent (Turco*) prior to decontamination of N Reactor.

Two 4000-gal batches and one 3000-gal batch of each waste were prepared to provide a total of 22,000 gallons of simulated waste.

3.2 DRY BLEND PREPARATION

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The dry blend was prepared at the DMRHF. The dry blend was tested at PNL prior to the pilot-scale test to determine the desired mix ratio for the test (see Section 4.3.1). The dry-blend formulation is listed in Table 3.3.

The dry blend for the pilot-scale test was transported to PNL in trailers with 1000-ft^3 capacities. Three trailers of dry blend were used during the test.

TABLE 3.3. Dry Blend Formulation

රා	Component	Weight Percent	
	Portland Cement, I-II	41	
lo	Flyash, ASTM Class F	40	
	Attapulgite Clay	11	
700	Indian Red Pottery Clay	8	

"3.3 SAMPLING DURING THE TEST

To statistically determine grout homogeneity, it was necessary to obtain many samples of cured grout, grout slurry, simulated PSW, and dry blend. This section describes the system that was developed to extract undisturbed cores of grout from the monolith. The frequency of sample collection is described, as well as tests planned for the samples.

3.3.1 Grout Core Sampler

In order to obtain representative samples of grout from the pilot-scale monolith, PNL designed and tested samplers to extract grout without requiring core-drilling. The final design used in the pilot-scale test is depicted in

Tradename of Purex Corporation.

Figure 3.1. The sampler consists of a PVC sample tube in a steel pipe. Two O-ring seals in the annulus at the base of the sampler prevent grout from entering the annulus. As the sampler is inserted into grout that has not set, grout flows into the sample tube.

The sampler was built with standard materials (2-in., Schedule 40 PVC pipe and 2.5-in., Schedule 10 carbon steel pipe). The system was sized to provide adequate sample size while minimizing disturbance to the grout as it was inserted. The grout cures in the PVC tube and around the steel pipe. After a

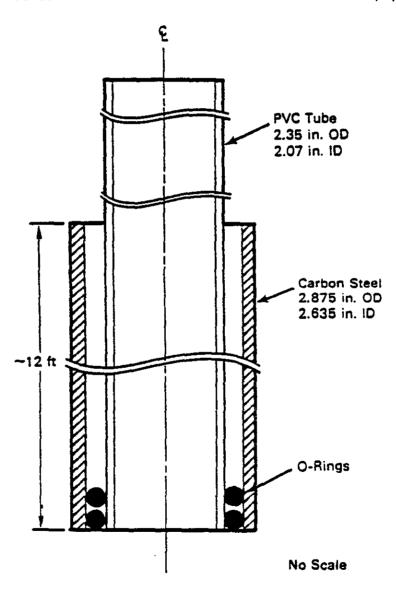


FIGURE 3.1. Grout Sampler Designed for Pilot-Scale Test

specified curing time, the PVC tube is lifted from the steel pipe and the grout remains in the PVC. Based on the expected tensile strength of the grout, less than 100 pounds of force should be required to fracture the grout at the end of the sampler in order to remove the PVC tube from the monolith.

Measurements taken after the samplers were removed from the monolith show a higher level of grout in some sampler tubes than expected. The level of grout in the samplers is expected to be the same level as the grout in the monolith that they were taken from. The fact that the levels of grout in the samplers were higher than expected implies that some upset to the grout occurred during insertion of the grout samplers. This factor may have some effect on the grout property measurements to be made on the grout in the samplers.

3.3.2 Sampling Plans and Tests

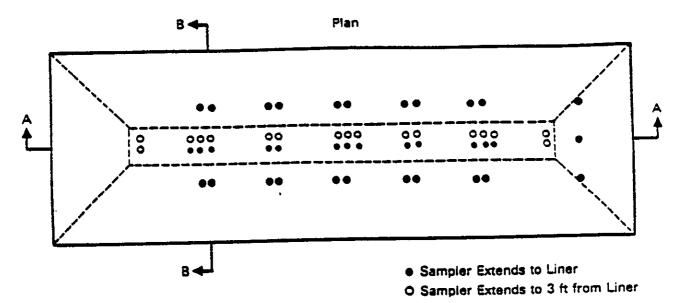
Sampling procedures for each type of sample were prepared prior to the test; operators were trained on their use. All sample containers were cleaned, dried, and sealed prior to the test. Sample containers were labeled and sealed as samples were taken. Operators taking the samples used chain of custody records and sample logs.

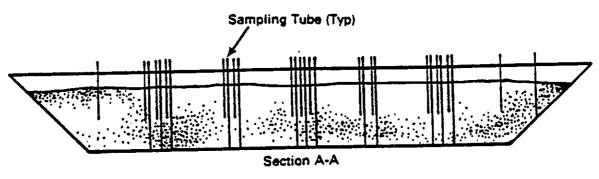
- The objective of the sampling plan was to obtain samples to:
- demonstrate that the grout monolith is homogenous
- provide information for design purposes (e.g., thermal properties)
- provide information for long-term performance assessment issues.

The sampling plan called for placing grout core samplers after 10 and 24 hours of grout production to obtain representative grout cores from the monolith. Figure 3.2 shows the locations of core samplers in the monolith.

The tests that will be performed on the grout cores include:

- leach tests EP Toxicity; Method 1310 (U.S., EPA 1982)
 - EPA toxic characteristic leaching procedure (when promulgated) (CFR 1986)
 - MCC-1 (Mendel 1985)





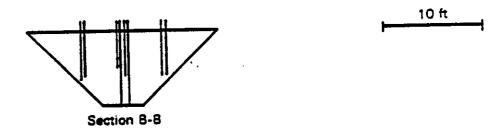


FIGURE 3.2. Location of Samplers in the Monolith

- ANS 16.1 (ANS 1984)
- oil and grease leachability Method 413.1 (U.S. EPA 1982)
- unconfined compressive strength; ASTM C-39 (ASTM 1985)
- capillarity; ASTM D2325 (ASTM 1985)

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- porosity/density; ASTM C-373 (ASTM 1985)
- thermal conductivity
- heat capacity
- compressibility; ASTM D2435 (ASTM 1985)
- composition
- corrosivity (WDOE 1984)

In addition, further tests will be performed with grout made in the laboratory using dry blend from the test:

- leach tests ANS 16.1 (ANS 1984)
 - MCC-1 (Mendel 1985)
 - TCLP (U.S.. EPA 1986)
- __ thermal conductivity
- co capillarity; ASTM D2325 (ASTM 1985)
- sulfate resistance; ASTM C452 (ASTM 1985)
- compressive strength; ASTM C39 (ASTM 1985)
- ___ compressibility/size stability
- porosity/density; ASTM C373 (ASTM 1985)
- heat of hydration
- thermal expansion.

In addition to the cores of cured grout obtained after the test, numerous samples were collected during the test at predetermined production times. Dry blend was sampled at the feed bin; subsequent analyses planned are X-ray diffraction (for determination of mineralogy), grain size, and cations (by inductively coupled plasma (ICP) spectrometry). Upstream of the mixer, PSW was sampled; its pH was measured immediately after sampling. Subsequent tests of the waste samples will include total suspended solids, EP toxicity, total organic carbon, and cation and anion analyses. Grout slurry was sampled at the

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mixer discharge and at the trench discharge. Tests conducted on the grout slurry included rheology, bleed water, sonic velocity, penetration resistance, and compressive strength. Bleed water samples were collected from the pilot-scale trench during the month following grout production. Bleed water was analyzed for EP toxicity, pH, total organic carbon, total oil and grease, anions, cations, and total organic carbon.

The results of most of the analyses of samples will be documented in a separate report.

4.0 RESULTS

This chapter presents results from the pilot-scale test to date. The results focus primarily on equipment performance, behavior of grout in the trench, and the rheology of the grout produced in the test. Detailed chemical analyses of grout, dry blend, and simulated waste will be documented in a subsequent report to be published in 1987, along with data on monolith homogeneity and cured grout properties.

4.1 SUMMARY STATISTICS

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Pilot-scale grout production was initiated at 9:11 a.m. on July 29, 1986.

Production ceased at 8:16 p.m. on July 30. Total production time during this test was 24 hours. About 7 hours of down time occurred in the first 12 hours of the test due to flooding of dry blend through the feeder and subsequent plugging of the continuous mixer. After 12 hours, the operation went more smoothly; however, occasional flooding of dry blend through the feeder caused down times of up to 30 minutes. Section 4.5.1 describes the flooding of dry blend in detail.

About 16,000 gallons of simulated waste and about 115,000 pounds of dry blend were used to produce about 22,000 gallons of grout. The volume of the grout was 38% greater than the volume of the PSW used to produce the grout. The average mix ratio was 7.2 pounds of dry blend per gallon of PSW. The average grout level in the trench was 6 feet.

The density of the grout slurry averaged 11.47 lb/gal (0.16 standard deviation). No significant difference was observed in the specific gravity of the grout at the surge tank and at the discharge from the piping to the trench (see Figure 4.1). It is concluded that insignificant deaeration of the grout occurred in the surge tank.

All samples (nearly 600) were taken at their scheduled times. All grout core samplers (a total of 53) were placed in the fresh grout at the planned times and sufficient core length was obtained in each sampler to produce the required samples for statistical determination of homogeneity and for other tests to resolve design, safety, and performance assessment issues.

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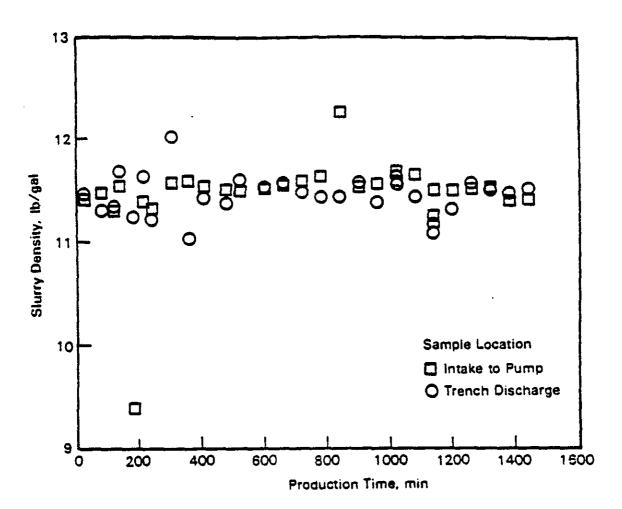


FIGURE 4.1. Specific Gravity of Grout at Surge Tank and at Discharge to Trench

4.2 BEHAVIOR OF GROUT IN THE TRENCH

Radioactive grout at Hanford will be pumped to underground concrete vaults for final disposal. Each vault will hold about 1.4 million gallons of grout. The planned interior dimensions of a grout vault are 125 feet long by 50 feet wide by 35 feet deep. The pilot-scale test provided valuable information as to how PSW grout will behave in a vault. Specifically, the test provided information on grout flow angles, temperature rise, separated liquid generation, setting characteristics, and degree of cracking. This section of the report presents the results of the pilot-scale test that pertain to the behavior of grout in the trench. (One topic not presented here is homogeneity, which will be covered in a subsequent report.)

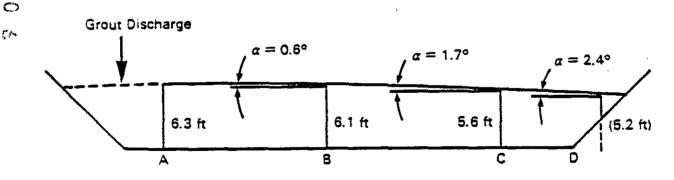
In this report, the end of the trench nearest the grout discharge point will be referred to as the high end. The end of the trench farthest from the discharge point will be referred to as the low end.

4.2.1 Flow Angle

In the proposed vault, grout will flow from the point of discharge out to 67 feet. Pilot-scale tests performed at PNL in 1984 and 1985 demonstrated that grout would fill a given space at a flow angle greater than zero (i.e., the grout is not self-leveling). The flow angle will affect the capacity of a vault; therefore, the flow angle required measurement on a larger scale.

The flow angle of the grout in the pilot-scale test is illustrated in Figure 4.2. The flow angle near the point where grout was discharged into the trench (the high end) was 0.6°. The flow angle increased to 2.4° at the low end of the trench. The average flow angle of grout from the discharge point in the trench to the farthest point (49 feet away) was 1.4°, corresponding to a 14-in. difference in grout depth.

In a test performed in May of 1986, 4000 gallons of grout poured into a 40-ft-long by 4-ft-wide trench exhibited an overall flow angle of 2° with some portions up to 3.5°. However, the mix ratio of this grout was higher (7.9 pounds of dry blend per gallon of waste) and the grout was visibly thicker. The grout flow angle in the disposal vault will be largely a function of the rheological properties of the grout as it is discharged to the vault.



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FIGURE 4.2. Grout Flow Angle in the Pilot-Scale Test

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Variations in the mix ratio and the degree of shear thickening that will occur in the piping will affect the flow angle. Because it is mandatory that the apparent viscosity of the grout at the discharge nozzle be low enough to maintain turbulent flow in the piping, such a grout should exhibit an acceptably low angle of flow.

To predict the flow angle of grout in a vault, several assumptions are required. The major assumption is that the rheological properties of TGF grout will be similar to those of the grout produced in the pilot-scale test. This assumption is reasonable in that the same dry blend, a chemically similar waste, and the same type of mixer and pump will be used. As discussed in Section 4.3.1. it will be necessary to control the mix ratio so that the grout flow rate exceeds the critical flow rate. A second assumption involves the extrapolation of the results of this test (in which the grout flowed 49 feet) to the actual case in which grout will flow 67 feet. As discussed in the next section, the grout flowed in thin sheets in relatively narrow channels that widened as the grout moved away from its point of addition to the trench. As the channel widens, the shear rate decreases. When the grout flows beneath separated liquid that collects on the surface of the grout, shear rate decreases further due to further lateral dispersion of the flow. As the shear rate decreases, the apparent viscosity increases, resulting in a greater flow angle.

Consequently, it is expected that the flow angles in the vaults will not be significantly greater than the flow angles observed in the pilot-scale test with grouts that have the same rheological properties. At the low end of a vault, a flow angle of 3° might be expected, leading to an overall flow angle of 2° or less. A flow angle of 2° corresponds to a 2.3-ft difference in elevation of grout from the center to the corners of a vault. For conservative design, the use of a 5° flow angle is recommended to provide contingency in the event that rheological properties vary significantly from those observed in the pilot-scale test.

4.2.2 Flow Patterns

No unusual grout flow patterns were observed in the pilot-scale test. The grout flowed in well-defined channels, with the channel width increasing with

increasing flow distance, but with decreasing definition. A typical channel width at the high end of the trench was 1.5 feet.

The pilot-scale trench was 4 feet wide at the base, widening gradually to the top. At the end of the test, the grout had risen to the 6-ft level where the trench width was 16 feet. (In contrast, the width of a disposal vault will be 50 feet.) The grout flowed in one channel until the level in that channel increased to the point that the stream diverted to a lower channel. Because the grout did not flow over the entire available area at any one point in time, the difference in trench and vault width is not expected to impact flow characteristics. Therefore, similar flow behavior is expected in a disposal vault. However, due to the greater production rate for grout disposal in the actual vaults, the grout will probably flow in wider, and perhaps deeper, exchannels at about the same velocity as observed in the pilot-scale test.

The grout produced in the pilot-scale test was thinner than that produced the test in May of 1986. As a consequence, the grout surface was quite smooth compared to the jagged surface observed in the May test. Figures 4.3 and 4.4 compare the grout surfaces in the two tests.

In the earlier tests, large, deep masses of flowing grout (as opposed to thin, layered flow at the surface) had been observed. This observation gave—rise to concern that the thermocouple bundles and/or the grout core samplers—could be displaced from their installed orientations. Therefore the bundles—and samplers were designed with anchors and braces to reduce this—possibility. In the pilot-scale test, no displacement of thermocouple bundles or samplers was observed. Additionally, trench observers did not note any massive movements of grout during the test. This is not to say that massive movements did not occur, however, because the presence of the trench cover restricted viewing.

One other concern was that samplers placed after 10 hours of production might interfere with grout flow. However, insignificant interference was noted. At the end of production, no flow lines or cracking due to flow disturbance created by the samplers was noted.



FIGURE 4.3. Grout Surface in Pilot-Scale Test

In summary, these flow pattern results indicate that grout will flow primarily in thin layers in well-defined channels that widen at increasing distances from the addition point. The grout surface is expected to be smooth if the critical flow characteristics match those of the grout produced in this test. If the vault cameras have enough resolution to observe the texture of



FIGURE 4.4. Grout Surface in Previous Test with Higher Mix Ratio

the grout surface, the TGF operators might be able to use this information as an indicator that the grout is too thick and that adjustments are required. If the vault design calls for level probes or other probes that penetrate into the grout, these devices should be anchored. Although massive movements of grout were not observed in this test, the potential for such movements exists.

4.2.3 Grout Setting

Tests performed in 1984 and 1985 with simulated PSW grout showed that grout that had flowed in smaller trenches (8 and 20 feet long, respectively) exhibited delayed setting at points furthest from the grout addition point. The cause of delayed setting was not understood, though the delayed-set grout exhibited a higher water content which could be related to the problem. If grout setting is delayed as a function of the distance that grout flows, then vault disposal design could be significantly affected. The pilot-scale test permitted evaluation of the rate of grout setting over flow distances approaching to those expected in a disposal vault.

In the 4000-gal test performed in May, grout that flowed 40 feet exhibited slightly lower set rates than grout near the addition point. However, the grout achieved penetration resistances exceeding 700 psi at all points within 7 days after production. Thus some of the concern of delayed setting was alleviated.

In the pilot-scale test, the simulated PSW grout set slightly slower at the low end of the trench. This slower setting was indicated by the ease of placement of the core samplers at the low end after grout production. However, the delay in setting was not significant enough to warrant concern regarding the vault design. It was not possible to quantify the strength of the grout via penetrometer measurements due to the high temperature and humidity in the trench. However, two days after production, the grout at the low end of the trench had developed enough strength so that a steel tube could not be pushed into the grout. Future compressive strength and penetration resistance tests on core samples will provide information as to the relative strength of the grout as a function of flow distance.

The delay in setting observed in prior tests can probably be explained by hydration kinetics and heat loss. The rate of hydration (and therefore the rate of strength development) increases with temperature. In previous smaller tests, the mass of grout undergoing hydration was much smaller than in the 4000-gal test and pilot-scale test. The heat of reaction was therefore more easily dissipated by the small trenches used in the smaller tests. The separated liquid that collected at the low end of trenches represented an

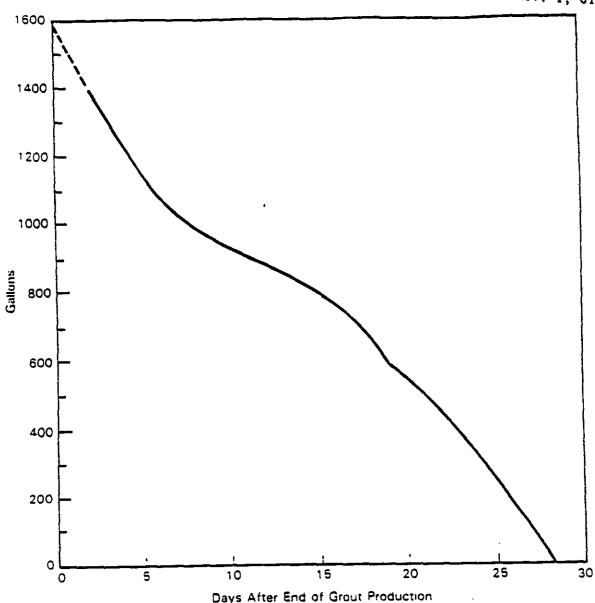
additional heat sink in both small and larger tests. Thus, lower temperatures explain why cure rates are slower at locations where separated liquid existed in smaller-scale tests. In a vault, the grout volume-to-vault surface area will be much lower than that of the pilot-scale test (8 ft versus 1.4 ft). Furthermore, the buried vault will be better insulated on all sides by the surrounding earth and cover. Therefore, grout temperatures and setting rates should be higher than those observed in the pilot-scale test. In summary, the pilot-scale test demonstrated that setting rates will be sufficiently high that the required compressive strength (50 psi) should be achieved at all points within several days following production.

4.2.4 Separated Liquid

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Separated liquid develops on the surface of PSW grout soon after the grout is produced. However, PSW grout was formulated to reabsorb all separated liquid within 28 days after production. Complete reabsorption occurs when the reference grout is produced in the laboratory and cast in small containers. However, in a large casting, separated liquid will pool in the low corners, such as in a vault. Because the separated liquid is not distributed over the entire surface of large castings, reabsorption may occur more slowly. The pilot-scale test permitted the evaluation of the amount and composition of separated liquid that can be expected in a sealed vault, and to determine whether the separated liquid will completely reabsorb.

The separated liquid in the pilot-scale trench covered approximately half the surface area of the monolith and accumulated in the end farthest from the discharge point. Figure 4.5 shows calculated volumes of separated liquid in the trench as a function of the number of days after production. About 1400 gallons of separated liquid were present in the trench two days after production. By extrapolation of data, as much as 1600 gallons may have been present immediately after production. Approximately 80 gallons of the separated liquid are attributed to flush water that was pumped to the trench during the production period and immediately thereafter. After 28 days, 27 gallons of separated liquid remained. All separated liquid was reabsorbed within 30 days.



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FIGURE 4.5. Volume of Separated Liquid

The decline in volume of separated liquid is primarily attributed to reabsorption by the grout. Undoubtedly, some liquid was lost by absorption into the wood cover and the soil in the anchor trench, by sampling, by vapor loss during viewing, and through minor leaks in the vapor barrier cover. It is estimated that these losses do not exceed 20 gallons, or 1.5% of the initial volume of separated liquid.

The reabsorption rate was highest during the first seven days after production (about 95 gallons per day). This corresponds to the period when grout temperatures were the highest and when the hydration rate was the greatest. After seven days, reabsorption rates subsequently fell to about 60 gallons per day.

Table 4.1 presents preliminary chemical analysis of the separated liquid as compared to the average synthetic waste feed analyses. The pH of the separated liquid is greater than that of the waste due to hydration of lime in the cement. Organic carbon, nitrate, and sodium are approximately the same concentration in both samples. Iron and phosphate are lower in the separated liquid due to precipitation of iron phosphate in the waste and incorporation of the precipitate in the grout. Sulfate, however, appears to concentrate in the separated liquid. More detailed analyses of separated liquid will be available in the future.

During production, a layer of immiscible fluid was floating on the separated liquid. However, this layer (perhaps containing tributyl phosphate) disappeared after five days. Apparently, it dissipated due to dispersion in the separated liquid. It is possible that as condensate refluxed in the trench and as liquid saturated with tributyl phosphate was being reabsorbed by the grout, the layer was totally dissolved.

TABLE 4.1. Comparison of the Compositions of PSW and Separated Liquid

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Item	PSW (With Precipitate)	Separated Liquid	
рН	12.2	13.1 - 13.2	
TOC, ppm	370 - 538	405 - 573	
SO ₄ , ppm	2,000	7,200	
PO ₄ , ppm	11,600	1,500 - 560 (decreasing with time)	
NO ₃ , ppm	400	350 - 380	
Na, ppm	12,600	11,000	
Fe, ppm	170	1	

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Based on results of the pilot-scale test, significant amounts of separated liquid can be expected in the grout vaults. The amount generated in the pilot-scale test was 7% of the grout volume. Laboratory measurements of grout made from a dry blend and waste from the pilot-scale test showed a separated liquid volume to grout volume from 15-16%. The difference in the laboratory grouts and pilot-scale grouts can be contributed to the type of mixing and the storage time of the dry blend. All the liquid in the pilot-scale test was reabsorbed into the grout in less than 30 days. Factors that will affect the amount of separated liquid in a vault include mix ratio, attapulgite characteristics, and flushing requirements. The removal of separated liquid should not be required if the separated liquid volume in a vault is less than 7% of the grout volume.

4.2.5 Cracking

The development of cracks in the grout is of interest because a high amount of cracking can significantly increase the surface area available for leaching, which could impair the ability of the grout to immobilize the waste.

The grout monolith was inspected on a regular basis to monitor crack development. The development of cracks was low in comparison with the level of cracking observed in the test performed in May. Cracks in the monolith were primarily parallel to the direction of the flow of the grout. The cracks appeared between the second and fourteenth day after the grout was produced and apparently grew little after they were first noticed. The maximum crack width appeared to be less than 0.25 inch. Cracks were most frequent at the high end of the trench.

Narrow cracks were observed on the fourteenth day after production, producing a crazed appearance on the grout surface. These cracks appeared to be less than 0.03 inches wide. They appear to cover the half of the monolith surface nearest the discharge nozzle.

The amount of cracking was much lower than observed in the 4000-gal test. In that test, significant surface cracking developed at the low end of the trench, but essentially no cracks appeared at the high end. In the pilot-scale test, cracks were most frequent near the grout addition point.

It is not clear why the severity of cracking was so much greater in the 4000-gal test. One possible explanation for the difference is that the lower mix ratio used in the pilot-scale test yielded grout with a smoother surface. Therefore, there were not as many stress points where cracks seemed to develop. Another explanation is that insufficient water was available in the 4000-gal test. However, because cracking was just as frequent in a portion of the trench that was kept under water at all times, it is doubtful that insufficient water was the cause for cracking. A third possible explanation was that at the low end of the 4000-gal trench, where the liquid level was close to or above the grout surface, the grout was not as dense. Therefore, cracks formed when shrinkage that accompanies curing occurred. However, this explanation is not supported by the pilot-scale test in which crack development at the low end of the trench was minimal.

The actual reason for the difference in cracking frequency between the two tests may be a combination of these explanations. However, the lower level of cracking observed in the pilot-scale test seems to be attributable to the lower mix ratio used. Thus, lower mix ratios seem to yield less cracking as well as lower angles of flow and lower potential for plugged lines.

Plans have been made to remove the monolith from the trench in FY 1987 and, in doing so, to further evaluate the degree of cracking that has occurred to provide data for assessing the long-term environmental performance of PSW grout.

0- 4.2.6 Temperature in the Monolith

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Temperatures at various locations in the monolith were monitored during grout emplacement and at least daily since then. Thermocouples were strategically located to permit the determination of the temperature rise in the monolith and the comparison of temperature profiles vertically, longitudinally, and laterally in the monolith.

The maximum temperature rise measured in the monolith was 37°C above the incoming grout temperature (29°C). Thus, the maximum temperature measured was 66°C at the high end of the trench near the grout addition point. Temperature profiles at the high end of the trench are shown in Figure 4.6. It can be seen

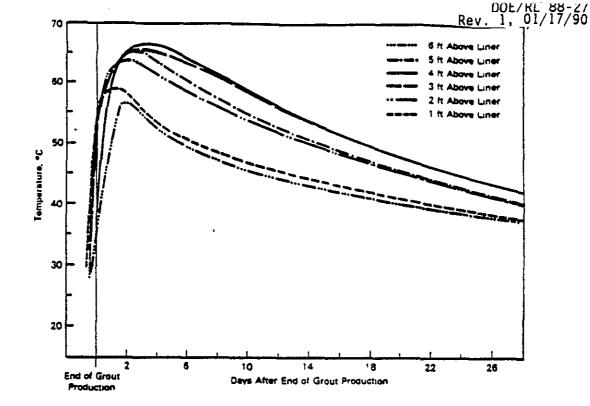


FIGURE 4.6. Temperature Profile - 2 ft From Discharge Nozzle

that the temperature peaked three days after the grout was produced. As expected, the middle depth of the monolith reached the highest temperature.

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Similar temperature profiles are provided in Figures 4.7 and 4.8 for the middle and low end of the trench. The profile 20 feet from the grout addition point is nearly identical to the profile at the addition point. In Figure 4.6 the thermocouple at the 6-ft level is obviously affected by the fluctuating ambient temperature in the trench, because the thermocouple was very near the surface of the grout.

The profile at the low end of the trench (40 feet from the addition point) is similar in shape to the other locations. However, the maximum temperature at this location is about 60°C. This lower temperature is probably due to the higher water content of the grout in this location as well as the smaller mass of the grout, which results in a higher rate of heat loss per mass of grout. For a comparison of temperatures longitudinally through the trench, refer to Figure 4.9, which depicts the temperature three feet above the liner at three trench locations. (The 3-ft level exhibited the greatest temperature rise in each of the positions.)

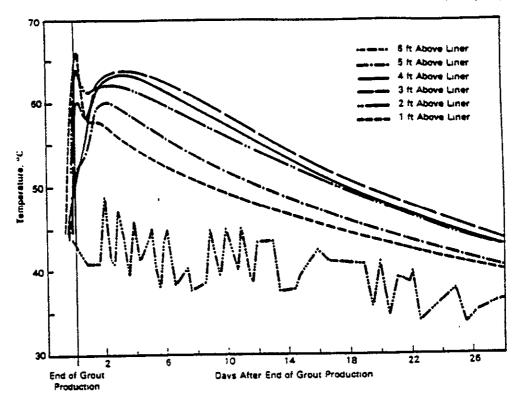


FIGURE 4.7. Temperature Profile - 17 ft From Discharge Nozzle

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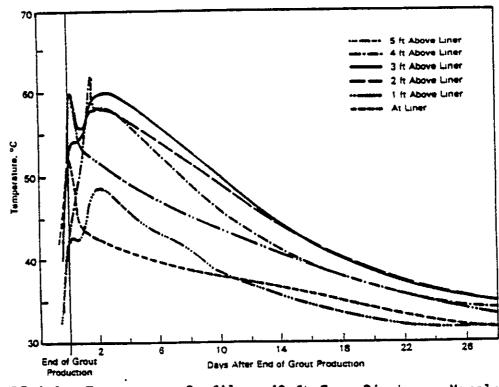
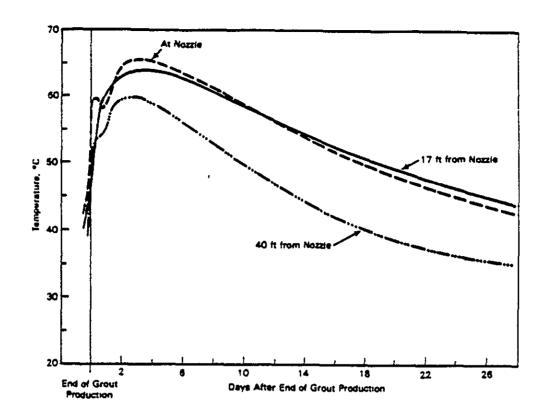


FIGURE 4.8. Temperature Profile - 40 ft From Discharge Nozzle



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FIGURE 4.9. Temperature 3 ft From Trench Floor at Three Trench Locations

Figure 4.10 shows isothermal contours in a cross-section of the trench 17 feet from the grout addition point. As expected, the temperature is dramatically lower near the liner and soil, which represent heat sinks. The lower temperatures at the grout surface indicate that most of the grout heat is lost at this location. The maximum temperature of grout observed within 2 inches from the liner was 54°C.

Lea (1971) presents heat of hydration data for portland Type II cement ranging from 46 to 61 cal/gram at 7 days. These data were determined through adiabatic tests and heat of solution tests. Using the maximum heat of hydration and heat capacities for the dry blend components reported by McDaniel, $^{(a)}$ an adiabatic temperature rise was calculated as a function of the water content

⁽a) Letter Report: McDaniel, E. W. et al. <u>Grout Formulation Studies with Hanford Facility Waste: An Executive Summary.</u> Oak Ridge National Laboratory, Oak Ridge, Tennessee (September 1984).

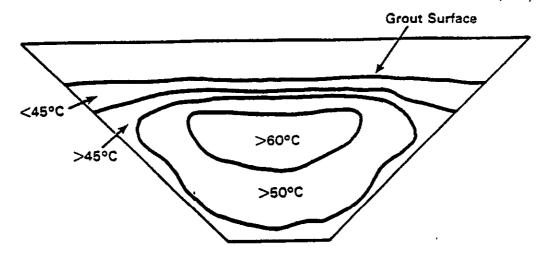
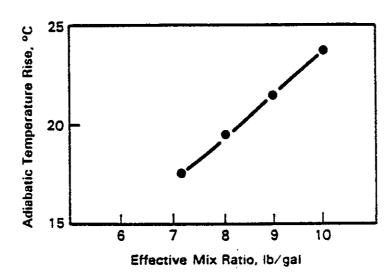


FIGURE 4.10. Isotherms in Pilot-Scale Trench Two Days After Production



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FIGURE 4.11. Theoretical Adiabatic Temperature Rise in Simulated PSW Grout as a Function of Mix Ratio (heat of hydration = 61 cal/g cement)

in the grout (Figure 4.11). As shown, the maximum temperature rise of 37°C measured in the pilot-scale test is considerably greater than the calculated rise at a mix ratio of 7.2 pounds of dry blend per gallon of waste. Even the temperature rise at the low end of the trench (31°C) exceeds the predicted temperature rise of 16°C. The difference in water content of the grout as a function of the position in the trench therefore does not explain the higher-than-expected temperature rise. It appears that more energy is released than

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expected from values reported in the literature. Possible explanations are:

1) additional exothermic reactions are occurring (e.g., flyash is reacting with lime), 2) the portland cement has a higher heat of hydration than reported in the literature, or 3) the portland cement rapidly and nearly completely hydrates in a few days in the grout environment. Based on the measured temperature rise and assuming adiabatic conditions existed in the center of the monolith, the calculated heat of reaction of the cement is 126 cal/gram.

Because of the discrepancy between the predicted and measured temperature rise in the pilot-scale test, laboratory calorimeter tests are recommended to establish the total heat of reaction in the grout mixture. Large-scale adiabatic tests are also suggested to positively establish maximum temperature rise and to provide data that can be compared with the laboratory test data.

Identification of the actual adiabatic temperature rise is required to determine the maximum temperature at which PSW can be fed to the grout process while ensuring that the upper temperature limit for grout will not be exceeded.

4.3 RHEOLOGICAL EVALUATIONS

Rheology is the field of study concerned with the deformation and flow behavior of materials. Viscous, pseudohomogeneous, multiphase fluid mixtures are classified according to their response to shearing stresses. PSW grout is classified as a pseudohomogeneous, time-dependent, non-Newtonian fluid (Lokken et al. 1986).

Prior to each test of the pilot-scale grout process, rheological and physical evaluations are performed on grout prepared in the laboratory using the simulated liquid waste and dry blend prepared for the test. These evaluations are used to determine the mix ratio to be used during the test that results in grout which meets established physical and rheological criteria. Lokken (a) has

⁽a) Letter Report: Lokken, R. O., P. F. C. Martin, M. A. Reimus, and C. J. Mann. 1986. Adequacy of Attapulgite Clays for Use in Hanford Facilities Waste Grouts. Pacific Northwest Laboratory, Richland, Washington.

shown that variability in attapulgite clay properties and in the blending process, for example, significantly affect grout slurry properties, and in turn, the mix ratio.

During each test of the pilot-scale process, rheological evaluations are performed to verify that the grout is in the turbulent flow regime throughout the entire transfer line. In addition, rheological evaluations are performed on grout from the process to determine if they are comparable to grouts produced in the laboratory.

In this section of this report, the method used to determine the mix ratio for the pilot-scale test is presented, as well as the results of rheological evaluations performed prior to and during the pilot-scale test. In addition, predictions of pressure drop in the piping are compared with actual data from the pilot-scale test.

4.3.1 Mix Ratio Determination

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The mix ratio (pounds of dry blend per gallon of waste) to be used in a \bigcirc test of the pilot-scale process is based on physical and rheological evaluations of laboratory-produced grouts mixed at different mix ratios. The mix ratios used in a pilot-scale test typically vary from 7 to 8 pounds of dry blend per gallon of waste.

The optimum mix ratio is the one that yields grout with the lowest critical flow rate, the lowest 10-min gel strength, and the lowest amount of drainable liquid. Compressive strength measurements at 28 days are also performed on selected grouts to verify that the strength exceeds 50 psi. PSW year, grouts produced with the reference formulation have compressive strengths 6 to 12 times the acceptable value of 50 psi.

In this section, the methods for measuring the critical flow rate, 10-min gel strength, drainable liquid, and compressive strength of the grout are presented.

4.3.1.1 Critical Flow Rate

The critical flow rate is defined as the flow rate at which turbulent flow begins. Grout must be pumped at rates sufficiently high to assure turbulent

flow at all locations within the pipe. Turbulent flow is required to prevent grout solids from depositing in the piping which could eventually cause plugging.

The critical flow rate is calculated using a critical Reynolds number of 2100, the power law model (Smith 1976), data from a Fann® viscometer, and the appropriate process parameters. For the TGF, the design critical flow rate is less than 65 gpm. For the pilot-scale test, the critical flow rate was required to be less than the operating flow rate of 15 gpm.

The use of a critical Reynolds number $(Re_{\rm C})$ of 2100 to calculate critical flow rate results in a nonconservative value for a non-Newtonian fluid such as grout. The use of a $Re_{\rm C}$ of 2100 is typically used for Newtonian fluids. The Metzner and Reed approach for pseudoplastic, non-Newtonian materials results in a critical Reynolds number of 2600 (Metzner and Reed 1955) for a typical PSW grout with the following properties:

flow behavior index (n) = 0.5 flow consistency index (K) = 0.127 lbf s/ft² density = 11.69 lb/gal

The critical flow rates associated with Re_c 's of 2100 and 2600 for a typical PSW grout in a 2-inch diameter pipe are 38 gpm and 44 gpm, respectively. Because the grout thickens in the pipe (discussed in Section 4.3.3), more conservatism is warranted in choosing the critical Reynolds number. Hence, the Metzner and Reed approach, where Re_c is based on the flow behavior index, is preferred.

4.3.1.2 <u>Drainable Liquid</u>

The amount of drainable liquid is determined by first pouring grout into a 250-mL graduated cylinder. During the next few days, the grout settles and drainable liquid appears on top of the grout. The volumes of solid and liquid are monitored periodically for 28 days. After filling the disposal vault, liquid that does not reabsorb will be pumped from the vault, stored in a tank,

[•] Fann Instrument Corporation, Houston, Texas.

and eventually processed into grout. This process is costly, therefore, it is desirable that the amount of drainable liquid be minimized.

4.3.1.3 Ten-Minute Gel Strength

The 10-min gel strength can be used to determine the theoretical maximum pressure the pump must apply to the grout to reinitiate flow following a 10-min downtime. When the grout is allowed to sit stagnant in a pipe, the grout will gel. To reinitiate flow, the pump must exert a pressure equal to the product of the gel strength and pipe surface area. Note that the gel strength needs to be known as a function of time; 10-min gel strength is an arbitrary choice to characterize grout gel properties.

The 10-min gel strength is determined after viscometer measurements have been conducted. The grout sample is allowed to sit undisturbed for 10 minutes in the Fann viscometer sample cup. After 10 minutes, the rotational speed is set at 3 rpm and the maximum dial deflection is read. The TGF specification for the 10-min gel strength is less than 100 lbf/100 ft 2 . Typical gel strengths of PSW grout range from 15 to 25 lbf/100 ft². A gel strength reading of 100 lbf/100 ft² corresponds to 100 lbf in 141 linear feet of 2-in. pipe. U Thus, for 1500 feet of pipe, a pump must be capable of generating 785 lbf to reinitiate grout of the specified gel strength.

4.3.1.4 Compressive Strength

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The current specification for grout compressive strength is a minimum of 50 psi. The compressive strength at 28 days is determined by pouring a sample of grout in a 2-in. diameter cylinder 4 inches long. The sample is sealed and allowed to cure undisturbed for 28 days. Compressive strength tests are conducted on an Instron® test machine in accordance with ASTM C-109 (ASTM 1985).

4.3.1.5 Summary of Grout Performance Criteria

In summary, acceptable mix ratios for PSW grouts are those that result in grouts that have critical flow rates of less than 65 gpm, minimal drainable liquids after 28 days, 10-minute gel strengths less than 100 16f/100 ft², and compressive strengths greater than 50 psi at 28 days.

Instron Corporation, Canton, Massachusetts.

Low critical flow rates are desirable because they result in: 1) lower flow angles in the grout disposal system, 2) lower temperature rise, and 3) perhaps a lower potential for grout cracking. Potential disadvantages of low critical flowrates include: 1) more separated liquid, 2) slower cure rates, and 3) less strength.

Laboratory tests and previous grout production tests have shown that grouts produced at mix ratios that result in critical flow rates of approximately 37 gpm (in TGF piping) possess the desired flow properties while still meeting the drainable liquid, 10-minute gel strength, and compressive strength criteria.

4.3.2 Pilot-Scale Test Rheological Evaluations

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Rheological evaluations of grout produced prior to and during the pilot-scale test were performed. The information gained from these evaluations and how the data were used during grout processing are presented in this section.

4.3.2.1 Tests Performed Prior to the Pilot-Scale Test

Prior to the pilot-scale test, laboratory tests were performed using the simulated PSW and dry blend prepared for the pilot-scale test to determine if a mix ratio of 7.5 pounds per gallon (the nominal mix ratio for the grout formulation) would result in a grout that would pass the critical flow rate criterion.

The critical flow rates of nine samples of grout produced in the laboratory averaged 10.4 gpm with a standard deviation of 0.37 gpm. These data indicated that turbulent flow would be achieved during the pilot-scale test if the targeted flow rate of 15 gpm was maintained. The grout was also expected to pass the drainable liquid and 10-minute gel strength criteria although these properties were not specifically evaluated due to the limited time available.

The critical flow rate calculated for grout produced in the laboratory prior to the pilot-scale test (mix ratio of 7.5) was lower than the critical flow rate for grout with the same mix ratio sampled from the pilot-scale process surge tank. The critical flow rates for the laboratory and process grouts were 10.4 gpm and 13.1 gpm, respectively.

Several factors affect the critical flow rate determined at a particular mix ratio. These factors include the amount of shear imparted by the mixing apparatus, the conditions at which the dry blend is stored, and the length of time it is stored. To negate the storage effects, rheological evaluations of laboratory grouts and process grouts need to be performed at the same time. Thus, laboratory mixing methods can be evaluated as to their effectiveness in duplicating the shear history imparted in the grout process.

At the TGF, it is planned to produce grout in the laboratory using actual waste samples and procedures that have been shown to be effective in duplicating the shear history of the process. Testing of such grouts will provide confidence that the predicted properties, such as critical flow rate, are valid for the expected processing conditions.

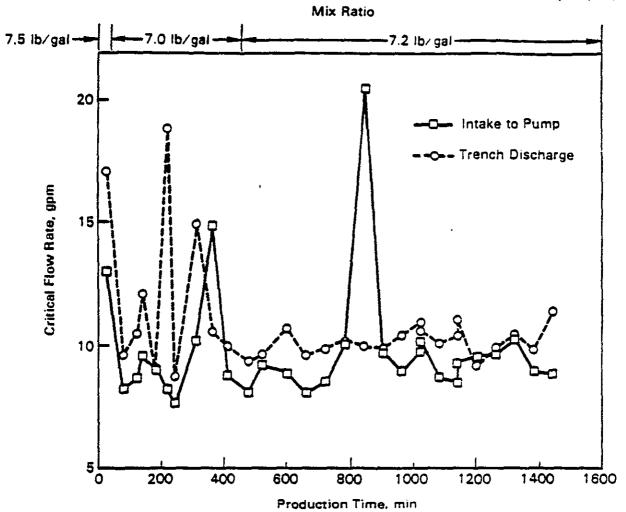
4.3.2.2 Tests Performed During the Pilot-Scale Test

During the pilot-scale test, critical flow rates for 1-in. sch 40 pipe (used in the pilot-scale test) were calculated based on grout properties at the surge tank and at the pipe discharge to the trench. The data were used to 1) indicate whether turbulent flow was maintained in the piping, 2) determine the effect of shear imparted by the flow of grout in the pipe, and 3) compare the properties of grout prepared in the laboratory to grout produced by the process equipment.

Figure 4.12 depicts the critical flow rates calculated throughout the test using the pilot-scale parameters. The mix ratio was adjusted twice during the pilot-scale test, once after the first 40 minutes when higher-than-desired critical flow rates at the discharge to the trench were measured, and once because changes were observed in the rheological properties of dry blend when a new trailer-load of the material was added to the process.

During the first 50 minutes of grout production, grout at the discharge into the trench was much thicker than at the surge tank. The shear imparted by pumping the grout through an equivalent length of 155 feet of pipe significantly thickened the grout.

The effects of shear thickening can be observed in the first time interval shown in Figure 4.12. The critical flow rate calculated at the surge tank was



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FIGURE 4.12. Pilot-Scale Test Critical Flow Rates at the Surge Tank and at the Discharge to the Trench

13.1 gpm whereas at the pipe discharge it was 17.1 gpm. Therefore, the mix ratio was decreased from the initial level of 7.5 to 7.0 pounds per gallon. Decreasing the mix ratio reduced the critical flow rate at the trench discharge to less than the operational flow rate of 15 gpm. Therefore, turbulent flow throughout the piping was assured.

The first dry-blend trailer change took place after approximately 400 minutes of grout production. Rheological data on grout produced with dry blend from the second trailer resulted in critical flow rates slightly less than

those calculated with the grout produced with dry blend from the first trailer. Therefore, the mix ratio was increased from 7 pounds per gallon to 7.2 pounds per gallon.

The second and final dry blend trailer change took place after approximately 975 minutes of grout production. The critical flow rates calculated from the grout made from the dry blend in this trailer were not significantly different from the previous critical flow rates. Therefore, no change in mix ratio was made as a result of this trailer change.

4.3.3 Shear Thickening Effects

Phosphate/Sulfate N Reactor Waste grout has been shown to be a pseudohomogeneous, non-Newtonian fluid sometimes exhibiting shear thickening properties (Lokken et al. 1986). The rheological properties of grout flowing can a pipe are dependent on the amount of shear induced by the flow in the pipe and by the amount of time the grout is subjected to that shear. The shear cainduced during pumping is a function of the velocity of grout in the pipe and the pipe diameter. For a given velocity, the shear rate induced by the 1-in.-diameter pipe used in the pilot-scale test is approximately twice that induced by the 2-in.-diameter pipe planned for the TGF.

except during one test in which the flow of grout was severely throttled through a nearly closed valve. In the 4000-gal test in May of 1986, shear thickening was observed by pumping grout through 71 feet of 3/4-in. pipe at 10 gpm. This was the first time dry blend from the DMRHF had been used in a test of the pilot-scale process. A difference in either the blending procedures and equipment used at the DMRHF and at PNL or the attapulgite properties is believed responsible for the observed shear thickening.

The effects of shear on the grout during pumping in the pilot-scale test can be evaluated from Figure 4.12 by noting the difference in the critical flow rates at the surge tank and at the pipe discharge. The average critical flow rate at the surge tank for the period when the mix ratio was 7.2 lb/gal was

9.25 gpm, whereas the average critical flow rate at the discharge to the trench was 10.24 gpm. Thus, the CFR increased approximately 11% in an equivalent length of pipe of 155 feet.

The TGF piping network is expected to cause less shear thickening per foot of pipe than experienced during the pilot-scale test (assuming identical grout properties at the pump discharge) because the shear rate will be about $340 \, \mathrm{s}^{-1}$ versus $600 \, \mathrm{s}^{-1}$ in the pilot-scale test. However, the TGF pipe network will be up to 20 times longer than the pipe network used in the pilot-scale test. The actual amount of shear thickening expected in the TGF can best be determined by pumping grout in a 2-in.-diameter pipe at the TGF flow rates over distances long enough to establish the effects of time at the appropriate shear rate. (This phenomenon will be examined in FY 1987.)

4.3.4 Pressure Drop Predictions

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The pressure drops expected in the 155 equivalent feet of 1-in. pipe in the pilot-scale test were calculated using the Metzner and Reed model, as described in Fow, McCarthy and Thornton (1986), and the Smith model (Smith 1976) for non-Newtonian, pseudoplastic fluids. The results of the pressure drop calculations and the data observed during the pilot-scale test are summarized in Table 4.2. The calculations were based on an average flowrate of

TABLE 4.2. Comparison of the Calculated Pressure Drops and Observed Pressure Drops

Grout Production Time, min	Calculated Pressure Drop-Surge Tank, psi		Calculated Pressure Drop-Pipe Discharge, psi		Observed Pressure Drop, psi
	M-R(a)	Smith(b)	M-R	Smith	
523	10.1	6.3	9.5	6.4	14.8
660	10.4	5.9	9.3	6.4	13.7
785	11.4	5.9 on Re 6.9 for Re	8.9	6.5	14.7
900	10.8	6.3	9.3	6.4	14.8
1254	11.1	6.4	9.3	6.4	14.7

⁽a) Metzner and Reed model (Fow, McCarthy and Thornton 1986).

⁽b) Smith model (Smith 1976).

15.3 gpm, an equivalent length of pipe of 155 feet, and a difference in elevation between the pump and pipe discharge of 149 inches.

The pressure drops reported in the second and third columns in Table 4.2 are based on the assumption that the grout rheological properties in the pipe did not change from those determined at the surge tank. In the fourth and fifth columns, the assumption that grout properties did not change from those determined at the piping discharge was used to calculate pressure drops. Theoretically, the pressure drop data measured during the pilot-scale test (Column 6) should lie somewhere between the predicted pressure drops in Columns 2 and 4 or 3 and 5.

In all cases, the pressure drops predicted by the Smith and the Metzner and Reed model were lower than those observed during the pilot-scale test. The Metzner and Reed model predicted 35% lower pressure drops whereas the Smith model predicted 55% lower pressure drops. For example, at 900 minutes of grout production, the pressure gauge at the pump discharge read 14.8 psi. The Metzner and Reed model using viscometer data generated with grout from the surge tank and from the pipe discharge into the trench predicted pressure drops of 10.8 and 9.3, respectively. The Smith model using the same viscometer data predicted pressure drops of 6.3 and 6.4, respectively.

The Smith model uses one curve for all non-Newtonian fluids to determine the friction factor at a given Reynolds number. In contrast, the friction factor from the Metzner and Reed model is dependent on the Reynolds number and the flow behavior index, n. The flow behavior index for the grouts reported in Table 4.2 ranges between 0.51 and 0.65. In addition, pipe roughness was taken into account in the calculated pressure drop using the Metzner-Reed model.

The Metzner and Reed model is recommended for predicting pressure drops for non-Newtonian, pseudoplastic fluids. However, viscometer data from the laboratory suggested that grout is actually a yield-pseudoplastic, (a) non-Newtonian fluid. In subsequent tests, more accurate rheological data can be obtained by using a Haake rotational viscometer or the Fann viscometer operated

⁽a) An explanation and discussion of yield-pseudoplastic fluids is found in Fow, McCarthy and Thornton (1986).

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at very low rpm's to determine a yield strength of the grout. Then, it is possible that more accurate pressure drops could be predicted.

4.4 FLUSHING

If grout stagnates in the pipe network or in the process equipment such as the mixer, pump, and surge tank, it will gel and eventually harden. The presence of hardened grout causes various problems, depending on the affected piece of equipment. To prevent the formation of hardened grout, routine flushes should be performed to remove grout accumulations from the equipment. In addition, flushes must also be performed when the processing equipment is shut down for more than a specified interval. (Twenty minutes was the interval specified for the pilot-scale equipment.) Because water is needed for flushing, but excess water is undesirable in the vault because it must eventually be removed, a compromise must be reached when designing the flush system and flushing procedures.

This section describes the flush systems for the pilot-scale mixer, pump, surge tank, and piping. Results of their effectiveness are presented. The improvements that were made to the pilot-scale equipment before the test to prevent accumulations of grout are discussed, as well as recommendations for further improvements.

4.4.1 <u>Mixer</u>

This section describes modifications made to the grout mixer to retrofit a flush system. The performance of the flush system is discussed, and recommendations are given for the flush system for the TGE mixer.

4.4.1.1 Modifications to Equipment

In previous tests, it was found that grout hardened in a 1/8-in. thick layer at the dry blend inlet of the pilot-scale mixer if the mixer was not flushed (Figure 4.13). Plugging of the mixer inlet could occur if the grout were allowed to accumulate during longer production periods. The pilot-scale mixer was subsequently modified with a spray nozzle in the dry blend inlet to spray a thin, cone-shaped spray of water down into the mixer.

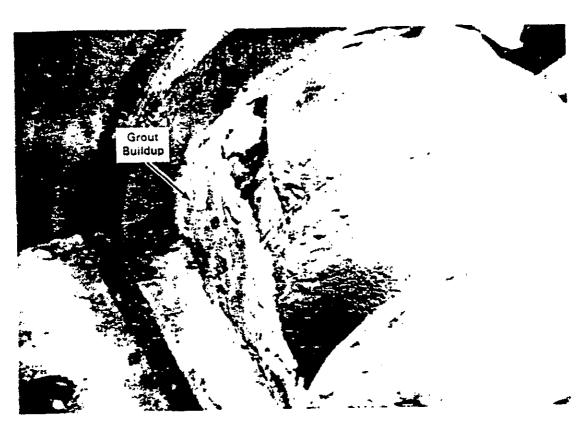


FIGURE 4.13. Grout Buildup at the Dry Blend Inlet Port Prior to Installation of Flushing System

4.4.1.2 Flushing Requirements

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The procedure for flushing the inlet of the pilot-scale mixer specifies flushing with water for 7 minutes at 1.5 gpm while the mixer is operating at 250 rpm. This procedure results in flushing the mixer with about 3.3 mixer volumes of water.

In the 4000-yal test performed in May of 1986, a single routine flush after 5 hours of grout production left an accumulation of about 0.1 inch of grout on the mixing blades. This accumulation was considered acceptable. In an effort to determine the maximum length of time allowable between flushes, routine flushes for the pilot-scale test were specified during the first trailer change (after about 10 hours of grout production) and every 12 grout production hours thereafter.

During the pilot-scale test, frequent downtimes necessitated flushing; therefore, routine flushes were not performed as scheduled. Instead, 10 flushes were performed after the system had been down for more than 20 minutes at a time. In seven of those flushes, flush water was discharged into a drum instead of the trench. This resulted in approximately 31 gallons of flush water discharged to the trench from flushing the mixer (approximately 30 gallons of water was flushed into drums).

4.4.1.3 Performance

Figure 4.14 shows the pilot-scale mixer before the test; Figures 4.15 and 4.16 show the mixer after the test. After the test, the mixer blades were coated with hard grout up to 1/8-in. thick. The dry blend inlet port was also coated with an accumulation of wetted dry blend and grout up to 1/2-in.

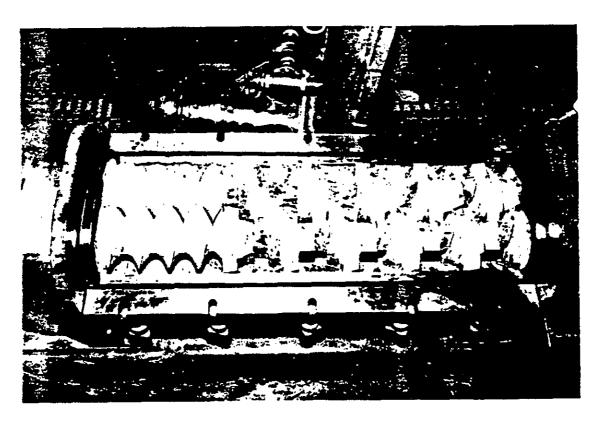
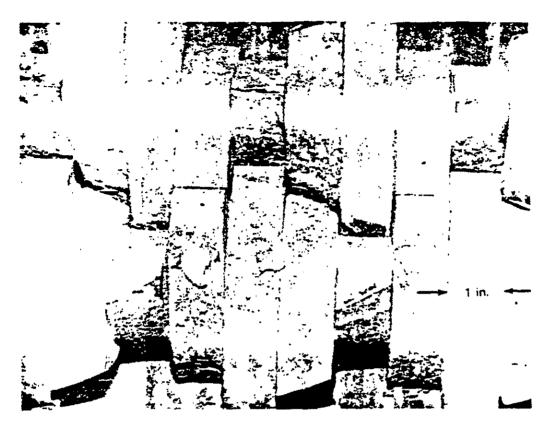


FIGURE 4.14. Pilot-Scale Mixer Before the Pilot-Scale Test



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FIGURE 4.15. Pilot-Scale Mixer After the Pilot-Scale Test

The buildup of grout on the mixer blades was probably caused by an inadequate number of flushes. The grout accumulated and hardened in the clearance spaces between the blades and the mixer cover (see Figure 4.15). Some wear of the plades was observed near the dry blend inlet port. This wear is discussed in more detail in Section 4.5.3.3.) The amount of grout buildup on the blades was limited because of the self-cleaning characteristics of the mixer blade design. More frequent flushing might have removed the grout before it had a chance to harden, which may have prevented some of the wear on the blades. The use of abrasion-resistant tips on the blades of the TGE mixer should also minimize wear.

The buildup of grout on the blades of the TGE mixer is not expected to be a major problem. Hard buildups on the blades may become dislodged, but the mixing action should reduce this dislodged buildup such that it can be pumped without causing damage.



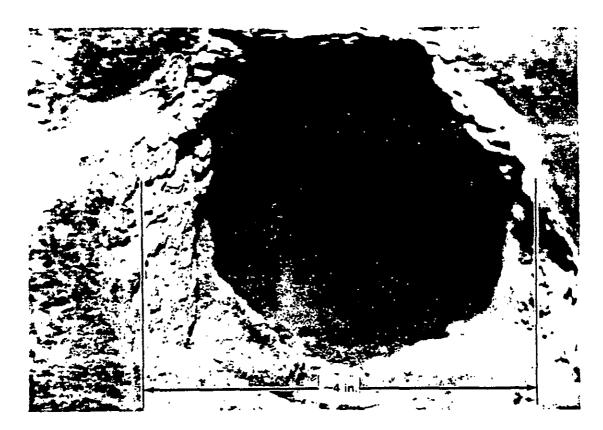


FIGURE 4.16. Dry Blend Inlet Port in Mixer Cover After the Pilot-Scale Test

The buildup of material in the mixer inlet is a more serious problem, however. Although the nozzle design for the flush system was effective in removing accumulations across the dry blend inlet port, it actually created a worse condition. Water from the nozzle contacted the screw section and was splashed up into the dry blend inlet port. During the pilot-scale test, the port was not allowed to dry before dry blend feeding was resumed. Consequently, the wet walls of the inlet port became coated with a layer of dry blend that hardened with time (Figure 4.16).

If the TGE mixer uses a flush system similar to that designed for the pilot-scale mixer, the dry blend inlet port should either be allowed to dry after flushing, or the inlet section should be constructed of a material that is not easily wetted.

4.4.2 Surye Tank

The surge tank was flushed manually with a hose. Water was delivered at flow rates between 5 and 8 gpm, using the least amount of water required to

clean the sides and bottom of the tank. The surge tank was flushed twice during the test. Flushing the surge tank contributed a total of about 20 gallons of water to the trench.

Though the TGE and pilot-scale surge tanks have similar residence times, the pilot-scale tank had areas where grout was stagnant. The surge tank for the TGE will be very different from the pilot-scale surge tank (see Section 3.5.4). If the tank is agitated as planned, grout solids should not settle out as they did in the pilot-scale surge tank. The TGE surge tank, as planned, will have a shorter residence time and have greater sloping sides (70° from the horizontal). The steeply sloped sides of the TGE surge tank also minimize the potential for grout to settle out on the sides and harden.

TGE designers must use care in sizing the tank agitator. The agitator must be effective in agitating the entire contents of the tank, but it must not impart so much shear that the grout thickens to the point that it cannot be pumped in turbulent flow. (The shear-thickening phenomenon is discussed in more detail in Section 4.3.3.)

4.4.3 Pump

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In this section, the modifications made to the pilot-scale grout pump to retrofit a flush system are presented. Also discussed are the flush requirements, the performance of the flush system, and recommendations for the flush system for the TGE grout pump.

4.4.3.1 Modifications to Equipment

In previous tests, a layer of hardened grout was found at the base of the inlet housing of the pilot-scale pump (see Figure 4.17). If the housing grout buildup were allowed to grow, as would be expected during a TGF campaign, the pump inlet could become plugged or large particles could break free and damage the pump. The pilot-scale pump flush system was designed to periodically flush out accumulations that may develop at the base of the inlet section.

The flush system included a flat-jet spray nozzle installed into the side of the pump inlet (see Figure 4.17). The flush water to the nozzle was controlled with a normally closed solenoid value that was automatically activated

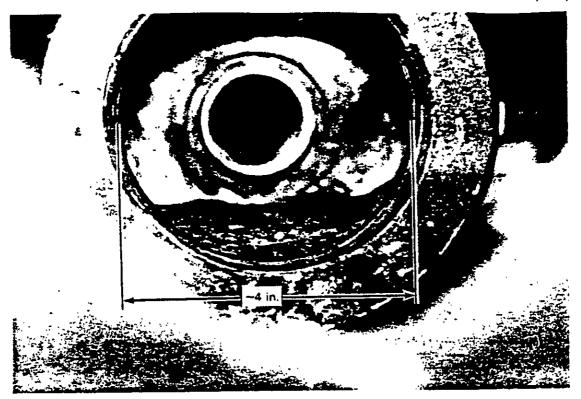


FIGURE 4.17. Cured Grout in Pump Inlet Without Flush System (Looking into pump inlet from discharge end)

for 3 seconds every 15 minutes. A total of 890 mL of flush water was delivered during each flush, corresponding to about 23 gallons of water used during the 24-hr test.

4.4.3.2 Performance

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Twelve hours after grout production ended, the piping to the pump inlet was removed. No hardened grout had formed on the base of the pump inlet. A few small chunks or "flakes" of cured grout were observed, however. These particles are believed to have fallen from the wall of the surge tank during the final flush at the end of grout production. It was apparent that the pump inlet flush system performed very well. A similar flush system is recommended for the TGE pump. However, the interval between the automatic flushes could probably be increased from 15 minutes to 30 minutes. The decrease in the frequency of the flushes would decrease the amount of flush water pumped to the

vaults. During a 1.4 million-gallon grout campaign, the pump inlet flush system operating at 30-min intervals would contribute about 200 gallons of flush water.

4.4.4 Piping

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To minimize the potential for grout accumulations in the piping, the piping to the vault should be designed to minimize the number of dead spots and sharp corners. Dead spots fill with settled grout solids, and, if not flushed clean, can eventually plug the line. To minimize the number of dead spots and erosion in the pipe, long radius elbows should be used whereever possible.

In this section, the flushing requirements for the pilot-scale piping are presented. Also discussed are the performance of the flushes as well as suggested flushing requirements for the TGF.

4.4.4.1 Flushing Requirements

The pilot-scale piping was flushed using the water from flushing the mixer and the surge tank. The water from flushing the mixer was discharged into the in surge tank. When the mixer flush was completed, the collected flush was pumped through the piping at a flow rate of about 11.5 gpm and at a Reynolds number of 35,000. Turbulent flow, which occurs at Reynolds numbers greater than about 2100 for flush water, is desirable to take advantage of the scrubbing effect. This procedure was repeated after the surge tank was flushed clean.

In the event the grout pump failed and could not be used for flushing the $_{\text{CN}}$ piping, the pump could be valved off at its discharge end. In such a case, water from a high-pressure pump was available to flush the discharge piping via a plug valve located near the pump discharge.

The pilot-scale test plan specified performing a routine flush after 10 hours of grout production, and then 12 grout production hours later. Because of process upsets during the test, the piping was flushed using the described procedure after four hours of grout production and not again until at the end of the run, 20 grout production hours later. In real time, this translates to the first flush being performed after 10 hours and the final flush 25 hours later.

4.4.4.2 Piping Performance

The amount of water flushed through the piping during each routine flush was equivalent to about 4.0 pipe volumes of water. Twelve hours after termination of grout production, the piping was disassembled and inspected for cleanliness. The first 125 feet of the piping looked very clean. A filmy buildup of grout (about 1/32-in.) had accumulated along the inside walls of the pipe. This buildup is not expected to be a problem because it will be scoured away the next time grout is pumped through the line.

The last 25 feet of pipe did contain accumulated grout. One horizontal section contained a buildup of soft grout that filled half the pipe (Figure 4.18). The fact that the grout in the pipe had not hardened after 12 hours suggests that the grout did not steadily accumulate in the pipe but instead was deposited near the end of the test. Records show that the pump speed was decreased near the end of the final flush as the water level in the surge tank was lowered to prevent running the pump dry. This action may have allowed solids to settle out of a solids-rich sluy of flush water.



FIGURE 4.18. Grout Buildup in a Section of Pilot-Scale Piping

The final flush water clearly contained a significant amount of solids due to the flushing of solids that had settled in the bottom of the surge tank. The excess buildup of solids in the run of pipe in Figure 4.18 may have been avoided if "clean" water had been flushed through the pipe following the final flush. To minimize the flush water added to the trench, this was not done. Another possible explanation for the solids in the last 25 feet of pipe is that this section of the pipe may have contained grout that was not in turbulent flow at all times. This condition may have resulted from the shear-thickening phenomenon previously discussed.

The TGE surge tank is not expected to accumulate grout solids as occurred in the pilot-scale surge tank. Cleaner flush water would result if no solids accumulated. If an adequate volume of relatively clean water is flushed through the piping at the end of processing, the amount of residual solids in the piping to the vault after flushing should be acceptable.

C. 4.4.5 Conclusions

The results from the pilot-scale test suggest that the flushing system for the pilot-scale mixer is inadequate. Consequently, recommendations cannot be made at this time for the flush system for the TGE mixer. The pilot-scale pump inlet flush system is satisfactory for application to the TGE pump although the interval between flushes could be increased to 30 minutes. The piping to the vault should be flushed with relatively clean water at a Reynolds number greater than 10,000. At least three pipe volumes of "clean" water should be used.

4.5 EQUIPMENT PERFORMANCE

One of the primary objectives of the pilot-scale test was to evaluate the performance of the pilot-scale grout processing equipment during an extended period of operation. Information on equipment performance can be used in the design of the TGF and in the preparation of TGF operating procedures.

In this section, the performance of the pilot-scale grout processing equipment used during the pilot-scale test is presented. Recommendations for improvements to the pilot-scale equipment and/or the TGF equipment are also discussed.

4.5.1 Dry Blend Transfer and Feed System

The dry blend transfer and feed system includes the supply trailer, the transfer system, the storage bin/baghouse, and the active bin/feeder (see Chapter 2.0 for details of the equipment).

Several problems were experienced with the dry blend transfer and feed equipment during the pilot-scale test. Occasionally, the dry blend would uncontrollably flood through the feeder, causing major process upsets. In addition, the high-level indicator and the vibrator in the storage bin intermittently failed to operate.

4.5.1.1 Flooding

Dry blend flooding caused major process upsets during the pilot-scale test. The first flood of dry blend occurred at the start of the test. Before the test began, the storage and active bin were emptied of dry blend that had been used in previous tests. To start the pilot-scale test, the storage bin was filled with fresh dry blend and then the active bin was filled. During the filling of the active bin, dry blend rushed through the feeder and out both the mixer discharge port and the oversize material port onto the vibrating screen. The fill valve between the storage and active bins was quickly closed, but not before approximately 20 cubic feet of blend had flooded through the system. This flooding incident was due to the flow of aerated and highly fluid dry blend through the feed pipe of the empty feed bin. The auger in the feed pipe did not provide a positive seal to prevent the discharge of fluidized dry blend.

During the test, significant flooding occurred ten times. Flooding would have occurred more often except that the operators learned to decrease the feed rate for a few seconds when thick grout was observed entering the surge tank. Flooding always occurred just after the end of a reload of the active bin.

Flooding of dry blend during the test was probably due to a vacuum leak around the butterfly valve located at the base of the storage bin. When the contents of the active bin are emptied to a preset level, that valve opens. This allows dry blend to fall from the storage bin to the active bin. It closes when the active bin is full. Soon after the valve closes, the blower is activated to convey dry blend from the trailer. This appears to be the time at which flooding occurred. The vacuum in the storage bin during the convey mode probably created vacuum in the feeder as it leaked around the butterfly valve. This probably resulted in fluidization of the material in the feeder bin, making it prone to flooding.

To compound the problem, the dry blend was transferred from the trailer up 42 feet to the storage bin through a 4-in.-diameter line. When the convey cycle shuts off, dry blend in the transfer line falls to the bottom of the line. This dry blend can temporarily plug the transfer line and cause greater vacuum at the onset of the convey mode, which can increase the potential for leakage through the butterfly valve.

To eliminate the flooding of dry blend, plans have been made to install a "bubble-tight" knife gate valve downstream of the butterfly valve. If, in the future, flooding does occur, an emergency shut-off valve to be installed—immediately downstream of the feeder discharge will be closed to stop the—flooding. This valve would be interlocked with the feeder such that the feeder auger could not turn if the valve were closed.

The pilot-scale test demonstrated the difficulty of handling and metering dry blend. Although the pilot-scale feed system significantly differs from the proposed TGF feeder, we recommend a thorough evaluation of the proposed TGF feed system for flooding potential, as well as thorough testing of the actual TGF feeder.

4.5.1.2 <u>High-Level Indicator</u>

The level sensor for the storage bin (a paddle-wheel type) is mounted on the side of the bin just below the baghouse. The level sensor is used to prevent overfilling of the storage bin. When dry blend reaches the paddle level, it creates enough torque on the paddle to stop the device from

turning. When the paddle stops, it sends a signal to the feeder controller to stop conveying dry material. During the test, the paddle wheel would occasionally stick, in which case the control system was given a false "full" signal and would not call for a transfer of dry blend from the trailer.

This problem was discovered when the active feed bin would only partially fill during a reload period. The faulty level sensor caused five short production interruptions, none of which required flushing of the grout-filled equipment. Based on this experience, the paddle wheel sensor cannot be recommended for the TGE application.

Alternative means of level sensing in the TGF dry blend feed system should be considered, e.g., load cells, capacitance methods, and vibrating level sensors. In subsequent tests of the pilot-scale process, plans have been made to replace the paddle-wheel level sensor with a vibrating level sensor. These vibrating sensors have been used extensively in dusty environments, specifically in fly ash and cement applications.

4.5.1.3 Bin Vibrator

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A vibrator on the storage bin was used to promote the transfer of dry blend from the storage bin into the active bin during a reload period. Occasionally the vibrator seized. Without the vibrator, the transfer of dry blend from the storage bin to the active bin was slow. It is desirable to fill the active bin rapidly to reduce the amount of time the feeder remains in a volumetric mode. Normally the feeder is operated in the gravimetric mode, which provides better control of the mix ratio.

The air to the vibrator was filtered but was not lubricated. To improve future performance, an oiler has been installed in the air supply line to the vibrator. A redundant vibrator will also be installed.

4.5.2 Vibrating Screen

A vibratory screen was installed upstream of the pilot-scale mixer to prevent oversized particles from entering and possibly damaging the mixer and the grout pump. During the pilot-scale test, the effluent from the vibrating screen was periodically weighed to determine the efficiency of the DMRHF in screening oversize particles. During 10 hours of grout production, 0.3 wt%

(0.5 vol%) of oversize particles was collected. Such weighing verifies that the DMRHF produces an acceptable dry blend for TGF operations.

4.5.3 <u>Mixer</u>

In general, the grout mixer performed very well. The following discussion analyzes the problems that occurred because of the dry blend flooding, the substantial dust generation, and the wear that was observed on a few of the mixer blades.

4.5.3.1 Dry Blend Flooding

At the start of the test, the discharge gate on the mixer was about 25% open to reduce dust generation from the mixer. When major flooding occurred, very thick grout and lumps of unwetted dry blend were produced. This overly thick grout caused a high torque on the mixer, which resulted in two shear pin rafailures. The mixer was cleaned out, the shear pin was replaced, and the test two started. The mixer shear pin is designed to fail at 20,000 in./lb, chefore significant damage to the mixer can occur. The TGE mixer will use motor to heaters instead of shear pins to prevent damage to the mixer.

As discussed in Section 4.5.1.1, the operators learned to avoid shear pin failure by reducing the dry-blend feed rate when thicker grout was observed and by stopping the mixer if thick grout continued to be produced.

When the mixer was flooded with dry blend, it was necessary to remove the comixer cover and manually remove the dry blend and thick grout. Such actions are not feasible in the TGE mixing module; therefore, reliable performance of the feed system is essential. Remote online viscometry at the TGE surge tank or other instrumentation at the feeder discharge might provide additional assurance that the TGE feed system is operating properly.

4.5.3.2 <u>Dust Generation</u>

Significant generation of dust occurred during the pilot-scale test. This magnitude of dusting had not been observed in previous tests when the vibrating screen directly upstream of the mixer was not in service. In the May, 1986 test, dusting was eliminated by partially closing the discharge gate on the mixer (75% closed). Because of the dry blend flooding during the pilot-scale

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test, a decision to keep the discharge gate fully open was made early into the test. A fully open gate was believed to be more capable of passing the thick grout produced during a flooding incident without plugging.

Dusting in the TGE mixer will be controlled by venting the surge tank to a filter system. Because of the potential for plugging, the use of the discharge gate is not recommended to control dust generation. Therefore, dust generated at the pilot-scale mixer in future tests will be exhausted by fans.

4.5.3.3 Equipment Wear

The screws and paddle blades immediately downstream of the dry-blend inlet port on the mixer showed some wear. A relatively thick coating of grout was observed on the top of the mixer lid where the blades passed. This grout layer is believed to be both the cause and result of wear observed on the screw and paddle blades. The grout layer would gradually increase in thickness as the blades were away.

The top of the screw blade eroded about 0.05 inch and the top of the paddle blade eroded about 0.2 inch. Figure 4.19 depicts the amount and location of the erosion on the blades. The blades in the pilot-scale mixer are made of 316 stainless steel. More frequent mixer flushes might have reduced the amount of wear observed. To reduce wear, stellite tips on the blades are recommended for the TGE mixer.

4.5.3.4 Mixer Efficiency

The grout mixer is intended to mix the dry blend and liquid waste, producing a very homogeneous slurry with a minimal amount of nondispersed particles. Mixer efficiency tests were performed during every 2 hours of grout production. A known volume of sample from the mixer discharge was poured onto a No. 30 screen. Water was gently run over the grout to wash away the slurry. The remaining particles were placed in a beaker and dried. After one day, the dry solids were weighed. The weight of the solids was divided by the volume of slurry to calculate mixer efficiency. The values of 11 mixer efficiency tests ranged between 0.56 grams of solids per liter of grout to 1.5 grams per liter (0.74 - 2.0 vol%). The average value was 0.96 grams per liter with a standard deviation of 0.33.

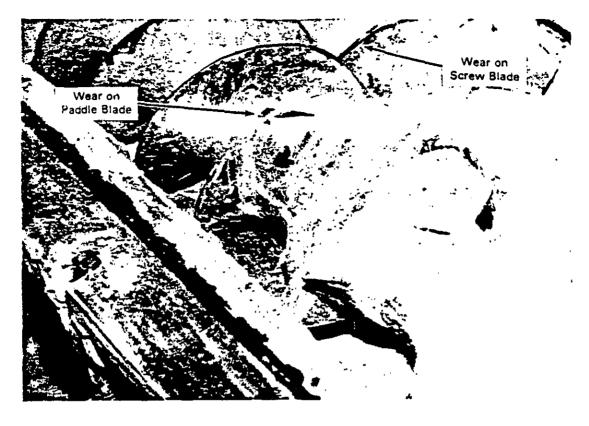


FIGURE 4.19. Wear on the Screw and Paddle Blade

In recent laboratory tests conducted at PNL to measure the effect of unmixed dry particles in grout, insignificant effects were found at dry particle levels up to 4 vol% (30 g/L). It is likely that the amount of unmixed particles in the grout is more a function of the quality of the dry blend than of the effectiveness of the mixer. In any case, dry blend from the DMRHF and the pilot-scale mixer produce grout of acceptable particle content. The same is expected of the TGE mixer.

4.5.4 Pump

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The progressive cavity pump performed satisfactorily. Prior to the test, a new stator had been installed; the pump with the new stator was calibrated with water before and after the test. This subsection presents the results of the calibrations and a discussion of the stator appearance after the test.

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4.5.4.1 Pump Calibrations

The pump was calibrated with water before and after the pilot-scale test to determine the wear on the stator after 24 hours of grout production. Results of the tests are presented in Figure 4.20. At 350 rpm and at 2 psi pressure head, the flow rate through the pump with the new stator was 15.5 gpm; the flow rate after 24 hours of grout production was 16.1 gpm. The difference in the flow rates is near the accuracy of the calibration method. Therefore, it is concluded that the stator experienced negligible wear.

Although minimal wear of the stator occurred in the pilot-scale test, results cannot be extrapolated with confidence to the TGE grout pump because pressures during TGE processing will be greater. The absence of a decline in performance over the 24-hr period of grout production is a positive indication that TGE pump life will be acceptable.

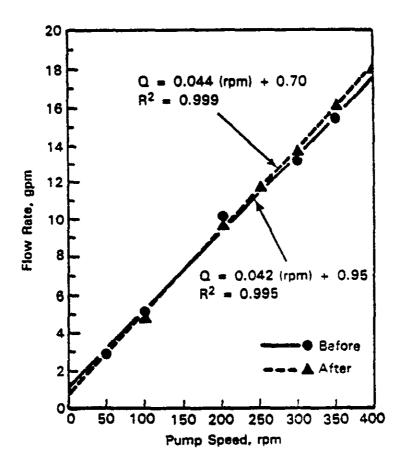


FIGURE 4.20. Pump Calibrations Before and After the Pilot-Scale Test

4.5.4.2 Stator Appearance

After the pilot-scale test, the pump stator was examined. Several circumferential delaminations, approximately 0.75 inch long and 1 inch deep, were observed in the discharge end of the stator. As of this writing, the stator has not been dissected to determine if delaminations are present inside the stator. Delaminations are not expected to occur in the TGF grout pump if a top-of-the-line stator is used.

4.5.5 Slurry Instrumentation

Process instrumentation for slurries in the pilot-scale test included the PSW flowmeter, the grout flowmeter, and grout pressure sensor. In earlier tests, a grout level detector in the surge tank was examined. All of the instruments performed satisfactorily except for the level detector in the surge tank. In this section, the performance of the process instruments for slurries is discussed. ('\ '

4.5.5.1 PSW Flowmeter

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The PSW flow rate was indicated by a rotometer and a magnetic flowmeter; the datalogger recorded the reading from the magnetic flowmeter. No problems were encountered with this system. A magnetic flowmeter with remote electronics to indicate the flowrate of radioactive LLW should perform satisfactorily in the TGF.

4.5.5.2 Grout Flowmeter

The grout flow rate was also measured with a magnetic flowmeter. The flowmeter performed satisfactorily, thus a magnetic flowmeter should also be acceptable for measuring grout flow rate in the piping to the vault.

4.5.5.3 Level Sensor in Surge Tank

The level sensor in the surge tank, a capacitance-type point sensor, was located near the bottom of the tank. The purpose of the level sensor is to warn the operator when the level of grout in the surge tank is low.

In prior tests, grout buildup on the sensor prevented the sensor from working properly. As a result, careful visual attention to the level of grout

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in the surge tank was required by the operator at all times. Based on this experience, a capacitance level sensor is not recommended for the TGE surge tank.

-4.5.5.4 Grout Pressure Sensor

The grout pressure sensor (a diaphragm type) was located immediately down-stream of the pump discharge. The sensor worked satisfactorily and is recommended for use in the piping to the vault. The pressure sensor is designed with a smooth, round surface so that flow is not restricted. Also, there are no stagnant areas where grout can build up and possibly plug the sensor.

In choosing a suitable pressure sensor for the TGE, several factors should be considered: 1) the effect of the level of radiation on the life of the material that contacts the grout and any fluid in the sensor, 2) the wear rate of the material that contacts the grout, and 3) possible dead spots where grout could build up and possibly plug the line. The type of sensor used in the pilot-scale test should be acceptable for at least low-dose waste.

4.5.6 <u>Trench</u>

A splash pad of 60-mil high-density polyethylene (HDPE) was placed directly below the discharge nozzle on top of the trench liner. To keep it in place, one corner of the pad was anchored with a steel plate. The splash pad was installed to protect the liner from possible abrasion due to splashing grout.

Although it is planned to recover and examine the splash pad when the monolith is exhumed, it is doubtful that conclusions can be extrapolated to a similar splash pad for the disposal vault, where grout will fall 35 feet to the vault floor. Therefore, a conservative design for the vault splash pag is recommended, e.g., a concrete or steel pad.

The trench cover performed as designed. The polyvinyl chloride (PVC) vapor barrier was effective in containing the moisture in the trench. The wood under the cover released some components onto the grout surface as evidenced by discoloration of grout directly under some of the joists. Cured grout properties will not be impacted because grout samples for analysis were kept

isolated by the PNL core sampler. It is possible that minor contamination of the separated liquid may have occurred, however.

Because the cover for the pilot-scale test bears no resemblance to a vault cover, no appropriate conclusions regarding the cover can be extrapolated to the vault design.

The discharge nozzle was merely an unrestricted opening of the 1-in. delivery pipe. The "nozzle" performed well; no spraying was observed as grout was discharged from the nozzle. Based on this experience, the open-pipe nozzle design appears acceptable for the vault application.

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5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

The objectives of the pilot-scale test performed on July 29 and 30, 1986, were successfully met. Data taken during and after the test were used to assess equipment performance and to evaluate grout behavior under conditions that closely approximate those expected in a vault. Nearly 600 samples of simulated PSW, dry blend, grout, and separated liquid were collected as specified in the test sampling plan. Several significant conclusions were drawn:

- The adiabatic temperature rise of a similar grout will be at least 37°C, and probably higher. The temperature rise of grout must be considered to ensure that the maximum grout temperatures do not exceed the evolving criteria.
- The maximum flow angle of PSW grout in a vault is not expected to exceed 3° for grouts with similar rheological properties. The average flow angle is not expected to exceed 2°.
- Separated liquid that forms on the surface of grout in a vault will probably be totally absorbed by the grout within 40 days after the termination of grout production, provided that the flush water pumped to the vault does not exceed 0.4% of the grout volume.
- The grout set within 2 days at all surface locations inspected. The faster-than-expected setting rate can be attributed to the accelerating effect of the relatively high temperatures achieved in the monolith. Similar setting rates can be expected in the vaults.
- Data collected during the pilot-scale tests show that the dry blend from the DMRHF has an insignificant amount of oversized particles.
- The pilot-scale grout mixer and pump, which are similar to those planned for the TGE, performed satisfactorily, as did most other components of the pilot-scale process. It is believed that

relatively minor changes in the process design are required to ensure reliable operations. Consequently, the TGF should also be capable of satisfactory operation.

• The degree of cracking of grout in the trench was minimal, reducing concern about how cracking would affect the performance assessment of this disposal method. (Cracking of a monolith creates additional surface area, which can lead to increased release of contaminants from the monolith.)

5.2 RECOMMENDATIONS

The results of the pilot-scale test indicate a need for additional analyses. Suggestions for the TGE design and modifications to the pilot-scale equipment are also provided.

5.2.1 Further Analyses

- Additional tests should be performed to determine the maximum temperature rise expected in the vaults.
- A study of grout mixing methods in the laboratory should be performed in conjunction with a pilot-scale test to establish a laboratory mixing procedure that yields grout that satisfactorily simulates grout produced with the pilot-scale equipment. This procedure would be used at the TGF with actual waste samples to verify grout processability and other properties prior to grouting specific batches of actual wastes (planner for FY 1987).
 - An experiment using a grout pump and piping similar to the TGF equipment should be performed to determine the amount of shear thickening expected in piping to the vault (planned for FY 1987).
 - A critical Reynolds number of 2600 should be used for more realistic calculations of critical flow rates of grout (Section 4.3.11).

5.2.2 Transportable Grout Facility

- Due to the flooding problems and consequences experienced in the pilot scale test, the TGF dry-blend feed system should be thoroughly evaluated for flooding potential. The TGF dry-blend feed system should also be tested under a variety of upset conditions before operation with actual radioactive grouts.
- The bearing housing on the discharge end of the grout mixer should be sealed to prevent bearing damage and/or contamination by grout.
- The TGF piping should be flushed with water at a Reynolds number greater than 10,000. Approximately three pipe volumes of clear water per flush should be used.
- An analysis of the impact of the shear imparted by the proposed TGE surge tank agitator on the grout should be performed.
- Paddle-wheel level sensors are not recommended in dusty environments;
 other level sensing devices should be considered.
- Stellite tips for the TGE mixer impellors are recommended to reduce wear.
- Capacitance level sensors in the grout surge tank of similar design to those used in the pilot-scale test are not recommended unless successfully demonstrated on pilot-scale equipment.

5.2.3 Pilot-Scale Equipment

- A bubble-tight knife gate valve will be installed downstream of the butterfly valve at the discharge of the storage bin to provide a better seal. (A poor seal was the suspected cause of dry blend flooding.)
- An emergency shut-off valve will be installed directly downstream of the feeder discharge. This valve will stop dry blend flooding should it occur.
- The paddle wheel high-level sensor in the storage bin will be replaced with vibrating high- and low-level sensors.

- A lubricator in the air supply line to the storage bin vibrator will be installed to improve vibrator reliability.
- Further development of the mixer flush system should be conducted.
- The bearing housing at the discharge of the mixer will be sealed to prevent grout from entering.

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